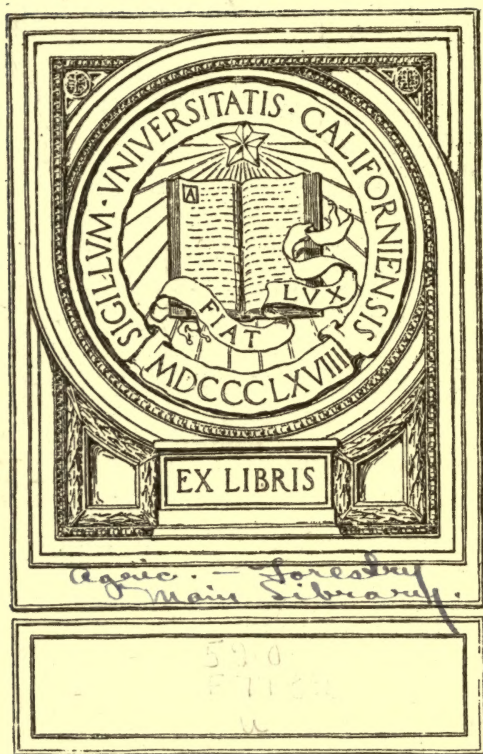




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UTILIZATION

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- 7 Suitability of Longleaf Pine for Paper Pulp. By Henry E. Surface and Robert E. Cooper. ^{USDA.} Bulletin No. 72.
- 8 Experiments with Jack Pine and Hemlock for Mechanical Pulp. ^{USFS.} By H. H. Thickens. 1912.

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- 9 Wood Turpentine. By L. F. Hawley. ^{F.S.} Bulletin No. 105.
- 10 Distillation of Resinous Wood by Saturated Steam. By L. F. Hawley and R. C. Palmer ^{F.S.} Bulletin No. 109.
- 11 The Specific Heat of Wood. By Frederick Dunlap. 1912. ^{F.S.} Bulletin 110. No. 252.
- 12 Wood Distillation. By W. C. Geer. ^{F.S.} Circular Industrial Alcohol: Uses and Statistics, No. 114. By H. W. Wiley, Chief, Bureau of Chemistry. Farmers' Bulletin No. 269.
- 13 Possibilities of Western Pines as a Source of Naval Stores. By H. S. Betts, 1912. ^{F.S.} Bulletin 116.
- 14 An Examination of the Oleoresins of Some Western Pines. By A. W. Schorger, 1913, ^{F.S.} Bulletin 119. Bulletin 516. 1912.
- 15 Yields from the Destructive Distillation of Certain Hardwoods. By L. F. Hawley and R. C. Palmer. ^{USDA} Bulletin No. 129.
- 16 The Analysis of Turpentine by Fractional Distillation with Steam. By Wm. C. Geer. ^{F.S.} Circular 152.
- 17 Rustic Wood: Its Substitutes and Adulterants By Geo. B. Sudworth and Clayton D. Mell 1911. ^{F.S.} Circular 184.
- 18 Quebracho Wood and its Substitutes. By

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Clayton D. Mell and Warren D. Brush. '12.

^{F.S.}
Circular 202.

¹ The Naval Stores Industry. By A. W.
Schorger and H. S. Betts. ^{U.S.D.A.} Bulletin
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²¹ Industrial Alcohol: Uses and Statistics/
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By A. Hugh Bryan and William F. Hub-
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Walter Mulford

United States Department of Agriculture,

BUREAU OF FORESTRY.—Circular No. 24.

A NEW METHOD OF TURPENTINE ORCHARDING.

The Bureau of Forestry will shortly issue a bulletin describing a new method of working Southern pines for turpentine. This method is intended to replace the present wasteful and injurious system of boxing. As the bulletin can not be published in time to reach turpentine operators before the present season's work begins, this circular is issued in advance for the purpose of giving operators an opportunity to install the new system, and thus avoid the loss in quantity and quality of products consequent upon another year of work with the old system of boxing.

The forthcoming bulletin will deal with the turpentine industry in general and with the economies that the new system makes possible. Here brief mention only can be made of the principal features of the system. Important among these is the production of 23 per cent more turpentine than by boxing and the yield of only the higher grades of rosin. The cost of the new equipment can easily be paid for from the extra returns of the first season, and there will also be an additional margin of profit. Moreover, since there is no box cutting under the new system, the least possible damage is done the trees; shallow chipping only is necessary.

For the season of 1902 the production of spirits of turpentine by the old system of boxing amounted to 600,000 barrels, valued at \$13,200,000. The production of rosin was 2,100,000 barrels, valued at \$4,200,000. The total value of the crop was thus \$17,400,000. Had the new method been applied to the same area of timber, the production of spirits of turpentine would have been 1,050,000 barrels, amounting in value to \$23,100,000. The production of rosin would have been 4,675,000 barrels, worth at current prices \$7,350,000. The total value of this product would have been \$30,450,000. This increased yield by the new system represents a net loss to the Southern naval stores industry of \$13,000,000 from boxing in one season.

It is important to note here also that the ordinary labor used for all boxing operations readily adapts itself without extra cost or training to the working of the new system.

This system was devised by Dr. Charles H. Herty, Expert in the Bureau of Forestry, and under his supervision received a thoroughly practi-

cal test at Ocilla, Ga., during the season of 1902. Its essential features are as follows:

1. An earthen cup of the same capacity as the standard box is used to catch the crude turpentine. At the top of the cup there is a stout rim, with a half-inch hole, by which the cup is suspended from a nail driven into the tree (fig. 1).

2. Two thin galvanized sheet-iron gutters convey the resin into the cup. These gutters are each 2 inches wide and from 6 to 12 inches

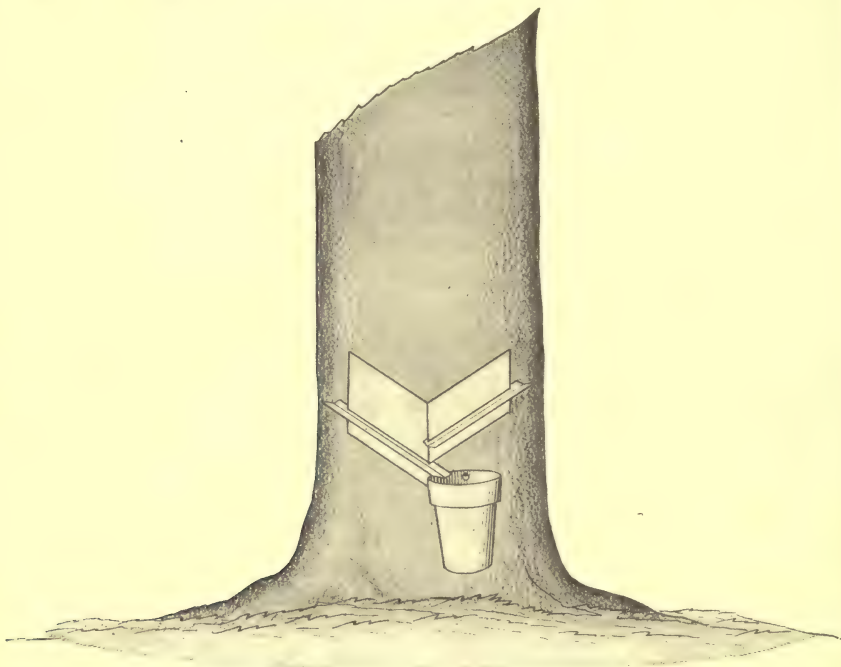


FIG. 1.—Showing the position of cup and gutters.

long, and are bent lengthwise through the middle at an angle of about 120° , thus forming an angled trough (fig. 3).

INSTALLING THE EQUIPMENT.

This system can be applied to trees previously boxed as well as to unboxed timber. In placing the equipment on unboxed timber, two flat faces are provided by means of cornering axes, a right-handed and a left-handed man working together. The first strokes with the axe are the same as in cornering the box. Next, by upward strokes of the ax, enough of the bark and sapwood is removed to form flat faces one-half as high as the distance between their outer edges. In this way the two men prepare a double face, corresponding in width with that of the box which would have been cut in such a tree.

Two inclined converging incisions are then made, one across each flat face, for the reception of the two sheet-iron gutters. The incisions are made by a right-handed and a left-handed workman, each with a broad-ax, having a straight-edged blade 12 inches wide. The men work on opposite sides of the tree; each man directs the head of his broadax somewhat downward, and with one stroke makes an inclined gash about one-fourth of an inch deep, across the flat face on his side of the tree. The incisions are made slightly deeper at their lower ends. The gash on one side should be made about an inch lower than on the other, but both should be so placed that there will be a space about 3 inches wide above them for the passage of the hack in the later chipping of the faces. Into the incisions thus made the galvanized iron gutters are firmly inserted, either by pressing one edge upward into the cut, or, better, by slipping the gutter endwise into the upper end of the cut and pushing it downward into place (fig. 1). The lower end of the upper gutter is brought to, and then forced down one-fourth of an inch beyond, the angle where the two faces meet; while the inner end of the lower gutter should pass beyond the angle about one and one-half inches, thus forming a spout to convey the resin from both gutters into the cup below (fig. 1).

In hanging the cup, care should be taken to drive the nail (a common 6-penny wire nail) at a sharp downward incline, and deep enough to leave only about three-fourths of an inch exposed. The nail is driven on the same side of the tree with the upper gutter, so that the cup when hung shall not be more than half an inch below the end of the spout of the lower gutter, and the nail shall be as far as possible from the dripping resin. This point is of great importance, for when the cups are filled with rain water, the fresh resin, lighter than water, at first forms a floating disc on the surface. As more resin drops on this, the disc enlarges until it touches and becomes anchored to the wall of the cup. If the cup is properly placed, this point of contact will be well away from the nail-hole, which will consequently not be clogged by the resin. As more resin drops upon this floating disc, the growing weight will shift the center of gravity and cause the viscous mass to sink lower along the side of the cup nearest the spout; at the same time, the water level will rise until the nail-hole permits an overflow. Thus each fresh drop of resin will force out a drop of water, while as the water does not rise to the top of the cup no resin is floated over the edge. After the streak has practically ceased running, the water above prevents the resin in the cup from losing its spirits of turpentine by evaporation.

Where the equipment is to be placed on trees which have previously been worked under the box system, it is unnecessary to provide the flat faces described, as the surface left by the chipping of the previous season answers the same purpose. One of the chief advantages of the new method is that the cup and gutters are placed each season immediately below the streak, thereby insuring a yield greater in quantity and

of better quality than is possible where the resin must flow over a long dry face to reach the box. On the higher faces the workmen with the broadaxes change sides, the blow being then delivered by an over-hand stroke.

The installation, in detail, is as follows:

On virgin, or unboxed, timber.—Three pairs of workmen (right-and-left-handed), with cornering axes, lead the squad, cutting the flat faces for the gutters. These are followed by a right-handed and a left-handed laborer, who make the incisions with broadaxes for the gutters. These eight men work back and forth across the drift in narrow lines and are

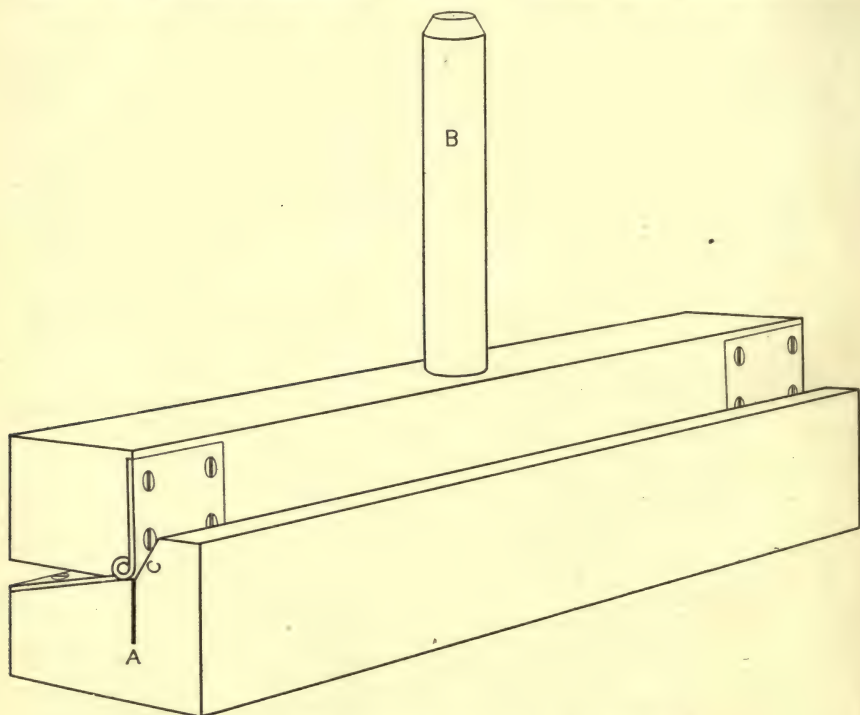


FIG. 2.—A device for bending gutters (one-third actual size).

followed by eight others, who quickly insert the gutters before the incisions begin to close, drive the nails, and hang the cups. In order that no time be lost, the cups should be distributed to each tree by a boy following closely after the axmen. In making the incisions with the broadax, care should be taken that the head of the ax is turned down only slightly, otherwise the inner half of the gutter may have too much slope, and upon the formation of the scrape in it the resin from a fresh chipping will run over the outer edge and be wasted. Two men are usually able to cut about 3,000 faces per day.

Two inspectors, one for the ax work and one for the gutter work, are

required. Tallying the faces is unnecessary, as an accurate record of these can be kept from the number of crates of cups used.

On boxed timber.—The six laborers with the cornering axes are unnecessary in applying this system to boxed timber, as the chipping of the previous season furnishes the flat faces required. The remainder of the work is therefore the same as on virgin, or unboxed, timber.

Whether the cup system is applied to virgin or to boxed timber, the best labor available should be assigned to the axes. If that work is well done, there will be no difficulty with the rest.

SPECIFICATIONS OF THE EQUIPMENT.

The following descriptions give in detail the essential features of the cups and gutters required by the new system.

CUPS.

The cups used are of clay, and are similar to flower pots. The form recommended is shown in fig. 1, and has the following dimensions:

	Inches.
Top ----- diameter inside ..	5½
Bottom ----- do -----	3½
Depth -----	7
Rim ----- deep -----	2

The bottom should be oval inside and flat outside, and the side walls one-fourth of an inch thick. A nail hole half an inch in diameter should be placed half an inch from the top of the rim. This cup has the same capacity as the standard box.

GUTTERS.

On account of the variable size of trees, and consequently of the width of the faces, it is best to purchase the gutter iron in long strips 2 inches wide, which can be subsequently cut into the desired lengths. For this purpose a pair of No. 8 tinner's snips can be used, but care should be taken to avoid turning the corner of the iron where the cut is first made. One thousand and eighty-six pounds of No. 29, or 2,050 pounds of No. 28, gage galvanized sheet iron will furnish gutters for one crop of 10,500 cups. The gutters commonly needed range from 6 to 12 inches long. After the iron is cut to the proper lengths, it is necessary to bend each piece to an angle of about 120° along its full length (fig. 3). No expensive apparatus is needed for this bending; a simple wooden machine (fig. 2) costing about 50 cents to make is sufficient. In operating this device, the flat piece of gutter iron is dropped into the narrow slit (A) and the handle (B) is pulled forward until the motion is stopped by the beveled edge (C). The bent gutter is then removed. A few minutes' practice enables a boy to bend from twenty to twenty-five gutters per minute.

Gutter boxes.—The gutters (6 to 12 inches long) are carried by the guttermen in boxes made with sloping sides and partitioned for the different lengths.

Placing the gutters.—The following precautions should be carefully observed in placing the gutters:

(1) The gutter must pass into the wood throughout the whole width of the face; otherwise there will be a leakage of resin behind it.

(2) The upper and lower gutters must be at least 1 inch apart at their inner ends on the angle of the faces so that there shall be no choking by the accumulated scrape.

(3) The inner end of the upper gutter should come to the angle at the middle of the faces and project not more than one-quarter of an inch beyond it.

(4) It is absolutely essential to the successful working of the apparatus that the lower gutter form a spout by extending about $1\frac{1}{2}$ inches beyond the angle at the middle of the faces.



FIG. 3.—Showing form of gutter (one-third actual size).

(5) Both gutters must be pushed deep into the wood at the angle of the faces.

TOOLS.

Cornering ax.—This ax, common on all turpentine places, is the best tool for cutting the flat faces for the gutters on virgin timber.

Broadax.—Two broadaxes with blades 12 inches wide with straight edges are to be set on short straight handles, one for a right-handed and one for a left-handed man.

On virgin timber a stock broadax, single-beveled, is used. On boxed timber a double-beveled broadax is preferable.

Claw hatchet.—This is the only tool required by the guttermen. It may be used in driving the gutter in case it sticks in the incisions, and answers also for driving the nails upon which the cups are hung and for the removal of bark, etc., in order to hang the cups properly.

OPERATING WITH THE EQUIPMENT.

Chipping.—This operation is precisely the same as in boxed timber. The upper gutter is placed about 3 inches below the chipping surface, thus giving abundant space for the passage of the hack.

Pulling.—Here again no change whatever is made in the usual prac-

tice on boxed timber; the puller passes easily between the gutter and the streak.

Dipping.—The tool used for dipping the cups is a steel blade 8 inches long, 2 inches wide at the lower end, and 4 inches at the upper end. This may be made from an old saw and fastened to a wooden handle extending a little over the upper end of the blade. Where the cups have oval bottoms, the lower end of the dipping knife should be rounded to fit them.

In dipping, the accumulated scrape is first loosened from the gutters by means of this dipping knife and pushed down into the cup. The cup is then removed from the nail and the resin is cut from the walls by a circular movement of the knife and emptied into the bucket.

Cost of equipment for one crop.

Cups (10,500) at 1¼ cents each	\$131.25
Gutter strips (1886 pounds galvanized iron, No. 29 gage, cut in 2-inch widths)	103.27
Cutting and shaping gutters	4.00
Nails (6-penny wire)	1.05
Freight charges (estimated)	30.00
Labor at trees	80.00
Total	\$349.57

The prices given are those at which responsible firms will at present furnish the material required. If the cups be placed on boxed timber the item of labor may be reduced to \$30, as the chipping of the previous season furnishes, without further labor, the flat surfaces for the gutters, which slide more readily into the incisions on old faces than into those on freshly exposed sapwood. The estimated freight charges are based upon a material reduction in freight rates recently offered by the principal railroads in the turpentine belt.^a

^aThe following railroads have agreed to haul the equipment as Class P matter in carload lots, 24,000 pounds minimum carload:

Atlantic Coast Line Railroad Company,
Central of Georgia Railway Company,
Georgia Railroad,
Georgia, Florida, and Alabama Railway Company,
Georgia, Southern, and Florida Railway Company,
Macon and Birmingham Railway Company,
Seaboard Airline Railway,
Southern Railway Company,
Western and Atlantic Railroad,
Wrightsville and Tennille Railroad Company.

This rate became effective on November 10, 1902.

ASSISTANCE IN THE FIELD.

In addition to the instructions here given, the Bureau of Forestry offers to all operators without charge the assistance of Dr. Charles H. Herty, who will personally direct in the field the installment of the new method. Applications for assistance should be directed to the Forester, Bureau of Forestry, U. S. Department of Agriculture, Washington, D. C.

GIFFORD PINCHOT,
Forester.

Approved:
JAMES WILSON,
Secretary.

WASHINGTON, D. C., *February 2, 1903.*

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U. S. DEPARTMENT OF AGRICULTURE,

BUREAU OF FORESTRY—Circular No. 34.

GIFFORD PINCHOT, Forester.

**PRACTICAL RESULTS OF THE CUP AND GUTTER
SYSTEM OF TURPENTINING.**

By CHARLES H. HERTY, Ph. D.

RECEIVED
SEP 21 1914
Division of Forestry
University of California

INTRODUCTION.

The cup and gutter system of collecting crude turpentine, proposed as a substitute for the box system commonly in use, was described at length in a former publication.^a The saving that it effected was also shown in the experimental tests, the results of which were given at the same time.

Since the publication of the bulletin, experimental and comparative tests have been carried on steadily, but only the results of a year's commercial test of the new system on the turpentine farm of Messrs. Powell, Bullard & Co., at Ocilla, Ga., have been published.

This circular is issued to show the practical results of three years' working of the new system, to call attention to several improvements that have been made in equipment and methods, and to point out several faults that have developed. All the comparative tests were made on similar half crops as described in Bulletin 40. Readers who are not familiar with turpentinizing are referred to that publication, but it may be said in general that in this system the resin is collected in a suitable vessel, preferably of hard burned clay, being caught and conducted to this vessel by inclined metal gutters inserted in shallow cuts in the tree.

The advantages claimed for the system were two: First, that it protects the tree against the destructive action of storms and fire; second, that it increases both the quality and the quantity of the product.

RESULTS.

Since the publication of Bulletin 40 the plot of timber there described as a "first-year crop" has been worked two years more, as a "second-year crop" and as a "third-year crop," complete records being kept of the yield from the "boxed" and "cupped" halves of the crop, together with careful studies of the condition of the trees in each.

Messrs. Powell, Bullard & Co. have courteously furnished the results of the second and third years of operation. Summaries of these figures are given in Tables I, II, and III. They show that the cupped trees yielded \$1,284.04 per crop, or over 30 per cent, more than the boxed trees.

^a Bulletin 40, Bureau of Forestry, U. S. Department of Agriculture.

TABLE I.—*Spirits of turpentine from half crops.*

Year.	Cups.			Boxes.			Excess from cupped half crop.	Net price per gallon at time of operation.	Value of cup excess.
	From dip.	From scrape.	Total.	From dip.	From scrape.	Total.			
	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Cents.</i>	
First.....	1,385.3	205.0	1,590.3	1,134.7	153.7	1,288.4	301.9	40	\$120.76
Second.....	1,103.5	165.0	1,268.5	705.2	226.6	931.8	336.7	45	151.52
Third.....	781.3	136.0	917.3	536.1	190.5	726.6	190.7	45	85.82
Total.....	3,270.1	506.0	3,776.1	2,376.0	570.8	2,946.8	829.3		358.10

TABLE II.—*Net sales of rosin from half crops.*

Year.	Cups.			Boxes.			Excess from cupped half crop.
	From dip.	From scrape.	Total.	From dip.	From scrape.	Total.	
First.....	\$401.72	\$47.72	\$449.44	\$328.40	\$35.53	\$363.93	\$85.51
Second.....	286.88	53.24	345.12	132.42	84.08	216.50	128.62
Third.....	212.60	61.65	274.25	124.76	79.70	204.46	69.79
Total.....	901.20	167.61	1,068.81	585.58	199.31	784.89	283.92

TABLE III.—*Summary of gain from cupped half crops.*

Year.	Spirits of turpentine.	Rosin.	Total.
First.....	\$120.76	\$85.51	\$206.27
Second.....	151.52	128.62	280.14
Third.....	85.82	69.79	155.61
Total.....	358.10	283.92	642.02

Total value of products from three years of operation.

Cupped half crop \$2,688.55
 Boxed half crop 2,046.53

Gain from cupped half crop 642.02=\$1,284.04 per crop.

CONDITION OF TREES.

At regular intervals during the three years of operation, careful study was made of the condition of the trees in each half of the crop. The results of these studies by years follow:

* TABLE IV.—*Record of down and of dead trees.*

	Number of trees blown down.		Number of trees dead.	
	Boxed.	Cupped.	Boxed.	Cupped.
In 1 year.....	8	3	35	16
In 2 years.....	60	34	139	83
In 3 years.....	78	44	217	150

Of the 44 trees blown down in the cupped half of the crop during the three years of operation, only 8 fell because they were being turpented. These 8 were small trees on which too deep incisions

had been cut by the broadax. Of the 78 trees blown down in the boxed half of the crop, the fall of 59 was due to the cutting of the boxes. The larger number of cupped trees blown down from causes other than the method of collecting the resin is due to the fact that many had doty hearts and were unfit for boxing, but just as well suited to cupping as sound trees. Many of these were snapped off above the turpentine face by a storm in the spring of 1903.

TABLE V.—*Productive surface lost from all causes.*

	Boxed trees.	Cupped trees.
	<i>Per cent.</i>	<i>Per cent.</i>
In 1 year.....	21.02	10.60
In 2 years.....	30.78	21.20
In 3 years.....	34.20	27.12

By far the greater part of this loss in both half crops was due to constantly increasing "dry face." It is evident that in the first year the box cutting was largely responsible for the wide disparity in the amount of dry face in the two half crops, but after the first year the chipping was the determining factor. In fact, the rate of increase of dry face after the first year is greater in the cupped than in the boxed trees, and is due, no doubt, to the higher average number of faces per tree in the cupped half. (See Bulletin 40, page 27.)

IMPROVEMENTS.

Since the cup and gutter system has been in use, a number of improvements in the equipment and in methods have been made.

Cups.—The quality of the cups has been materially improved. Many of those first used allowed the resin to seep through. This was due to the fact that the cups were made in molds, a method of manufacture requiring a soft, wet clay. This objection has been entirely overcome by the use of machinery and a stiff and much drier clay. The new cups allow no seepage and are just as desirable for collecting the resin as if they had been glazed at considerable cost.

Shipping the cups.—The complete success attained in shipping the cups in bulk has effected a saving to the operator, both in decreased breakage in transit and in the cost of the wooden crates formerly used.

Dipping.—At the time cups were introduced it was hoped that with experience the dippers would be able to collect as much gum per day from cups as is usual from boxes. This hope has been more than realized, for in practically all cases it has been found that the dippers are able to collect more gum per day from cups than from boxes. Difficulty was frequently experienced in getting good box dippers to undertake the dipping of cups, but after the change was once made a preference for cup dipping was uniformly noticed.

Use of the broadax for facing.—The most important advance made in the application of the cup and gutter system has been the substitution of the broadax for the club ax in making the flat faces on the trees. The difficulty in using the cornering ax recommended in Bulletin 40, on the hard timber near the Gulf coast in south Florida, led to the suggestion by Mr. R. M. Radford, of Braidentown, Fla., that the broadax be used for making the faces. The experiment was tried, and the result was faster and better work and a good surface for beginning chipping. Later, Mr. H. H. Ellarbee, of Ellarbee, Fla., suggested that the usual setting of the broadax on the handle be reversed, so that in hewing the beveled side would be next to the tree. This change greatly increased the speed of the work, as the chip could be promptly split off after the full width of the face had been obtained.



FIG. 1.—Making the first half of a face.

The next step was to have one man make both incisions for the gutters instead of employing a right-handed and a left-handed ax-man for making the two. At the outset the laborers usually insist that one man can not make both cuts, but a little practice gives perfect facility in cutting each with equal ease.

These changes have made it easily possible to double the speed of a squad, and many laborers have become very skillful in this use of the broadax. One laborer during the past winter prepared in one day 1,700 faces. Inspection of the work shows that it was well done in every respect.

Directions for using the broadax.—The ax should weigh from 8 to 9 pounds and have a perfectly straight edge. The handle should be straight and not longer than 30 inches. Each laborer sets his ax on the handle so that its edge is parallel to the handle and the beveled side lies next to the tree in hewing.

By almost vertical strokes of the ax a flat surface is hewn one-half the width of the "face" which is to be chipped later (fig. 1). When the ax first catches the wood the chip is prized outward slightly, thus facilitating the easy entrance of the ax in the same cut on the second stroke and also keeping the surface smooth. On the third stroke it usually is possible to partly cut and partly break off the chip, thus avoiding useless mutilation of the tree. This is done by a twist of the

ax, given just as it falls into the previous cut, the eye of the ax being thrown against the tree and the edge outward. The laborer then steps either forward or backward and hews the second side of the angular face (fig. 2), taking care to leave no rounded surface in the center where the two sides join.

On these flat surfaces the incisions for the gutters are then made, one a right-handed and the other a left-handed cut (fig. 3). In making these cuts the ax is held so that the outer point of the edge is lower than the inner point, while the eye of the ax is lower than the edge. By a single blow a cut is made the full width of one side of the face, this cut being about one-quarter of an inch deep at the outer edge of the face and a little deeper at the center. The cuts should be at least one inch apart at the center of the face, to prevent choking between the two gutters by chips and "scrape," and for convenience in cleaning the gutters when the cups are dipped.

Zinc nails.—The cup and gutter system has been most favorably received by the owners of mills where the timber is sawed after it has been turpentine. The only objection they make is to the use of iron nails for hanging the cups. To meet this objection zinc nails are now manufactured for use with the cups. Experiments have shown that if such a nail is left in the tree no damage whatever is done to a saw that strikes it, because the metal is so soft.



FIG. 2.—Making the second half of a face.



FIG. 3.—Making incisions for the gutters.

ERRORS MADE IN USING THE CUP AND GUTTER SYSTEM.

Gutters.—In making the incisions for the gutters the eye of the broadax is frequently turned down too much. The outer half of a gutter inserted in such an incision lies almost at a right angle to the trunk of the tree, and hence forms a shelf rather than a trough (fig. 5, B). From such a gutter the resin frequently runs off along the outer edge. Furthermore, such a gutter is easily forced out when the dipper is removing

the "scrape" from it, the pressure of the dip knife being almost in line with the direction of the cut. A gutter inserted carefully into a properly made incision will never fall out (fig. 5, A).

Nails.—Two mistakes are frequently made in driving the nail on which the cup hangs. First, the nail is driven horizontally. In the latter part of the season, when hardened resin collects under the head of the nail, a cup hung on it will easily drop off. The head of the nail should have a steep slant upward. A cup hung on a nail so driven fits snugly between the nail and the tree, is secure, and can not swing away from the spout of the lower gutter, though it may be easily removed by the dipper. Second, the nail is often driven into the tree above the lower end of the lower gutter. A cup hung on such a nail is never vertical, and its capacity is therefore diminished. Further, when a cup so hung fills with water or resin the increased weight tends



FIG. 4.—Cup and gutters properly placed.

to bring it to a vertical position, thereby pressing on the lower gutter and causing the latter to be prized out of the incision. The nail should always be driven into the tree at a point slightly lower than the lower end of the lower gutter (fig. 4).

Cups.—Experience has demonstrated that the cups will be broken if water is frozen in them. During the winter, therefore, when the cups are not in use, they should be removed from the nails and inverted at the base of the trees, so that water can not collect in them.

FIXING LOOSENED GUTTERS.

In case a gutter is displaced during the chipping season, the accident can be easily repaired by reinserting it in the old incision and driving two nails into the tree flush with the bottom of the gutter—one nail near the center of the face and the other near the upper end of the gutter. Supported by these two nails a gutter will stand the scraping of the dip knife without slipping. The gutter and both nails can readily be drawn from the tree at the end of the season.

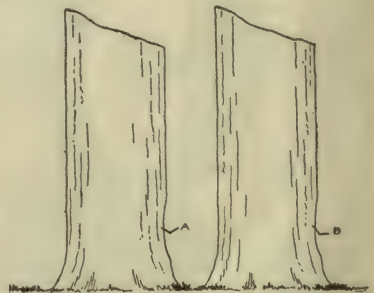


FIG. 5.—Position of gutters on the face.
A, correct; B, incorrect.

APPRECIATION OF THE NEW SYSTEM.

The rapid introduction of the cup and gutter system by turpentine operators proves beyond a doubt that the experimental stage has been passed. No stronger confirmation of the value of the results obtained at Ocilla, Ga., could be given than the fact that by far the greater portion of the cups placed during the past winter were on farms where the cup had been tested on a small scale during the preceding season. Not all operators are yet convinced; there are still doubters and scoffers, but their number is rapidly diminishing.

In the hands of a few the system has been abused. The fact that under it the trees are not weakened and made subject to overthrow by windstorms, as boxed trees are, has induced some operators to largely increase the number of cups per tree as compared with the average of boxes. The returns from such trees prove that there is a distinct limit to the sap surface which can be removed without weakening the vitality of the tree and lessening the flow of resin. The practice of overcupping results, therefore, in a financial loss.

In addition to the increased returns from the use of cups, the naval-stores industry has been benefited in two other lines by the development of the cup system. In many cases timber owners have made decided concessions in their leases to operators, on condition that the cups be used and no boxes cut. In other instances large tracts of timber have been brought under turpentine operation by the cup system, though their owners have persistently refused to lease them for box cutting.

The rapid rise in the value of turpentine timber within the past few years has contributed largely to its more conservative treatment. A thing of little value in the past, it has received only a small amount of care. Its rapid enhancement in value, and the strong probability of a still further increase, gives the turpentine operator every reason to treat his timber carefully. Formerly it was an easy matter for an operator to work hastily through a place, then move a short distance and develop a new place on timber just as inexpensive as that on which he had been working. But now the operator knows that when his present location is exhausted he is certain to experience difficulty in finding a new location, and equally certain that a largely increased price must be paid for it. This natural operation of the law of supply and demand is rapidly bringing serious and earnest thought to the question of how best to preserve the present holdings. The cup and gutter system with its great economies is doing much to solve the problem.

Approved:

W. M. HAYS, *Acting Secretary.*

WASHINGTON, May 27, 1905.

INVESTIGATIONS ON THE OIL OF EUCALYPTUS GLOBULUS OF CALIFORNIA

By CHARLES E. BURKE AND CHARLES C. SCALIONE

Received December 21, 1914

For many years considerable interest has been taken in California, in the *Eucalyptus globulus* (Blue gum), a tree which, while a native of Australia, has been transplanted and thrives well on many parts of the Pacific Coast. The fact that the tree grows very fast, and yields a hard wood makes it a tree particularly desirable in these localities which are almost destitute of hard woods.

Many difficulties have been met in seasoning the wood which has a great tendency to warp and check, but these have been largely overcome by experienced lumbermen and it is now easily possible with certain precautions to prepare from the *Eucalyptus* a good



FIG. I.—EUCALYPTUS GROVE ON THE CAMPUS OF THE UNIVERSITY OF CALIFORNIA. TREES APPROXIMATELY THIRTY YEARS OLD

clean lumber, which for hardness and tensile strength compares very favorably with oak or hickory.

In Australia one of the valuable products of the tree has been the oil which is obtained by the distillation of the leaves; this oil finds extensive use in the arts and in medicine and is imported in large quantities annually from Australia into the United States. The United States Pharmacopeia describes the oil as:

"A colorless or pale yellow oil, having a character-

istic or aromatic and somewhat camphoraceous odor, and a pungent, spicy, and cooling taste. Specific gravity, 0.905 to 0.925 at 25° C. Soluble in all proportions in alcohol; also soluble in three volumes of 70 per cent alcohol. Its alcoholic solution should be neutral to litmus. It is dextrogyrate, the angle of rotation being not more than +10° in a 100 mm. tube at a temperature of 25° C. If 2 cc. of the oil be mixed with 4 cc. of glacial acetic acid and 3 cc. of a saturated solution of sodium nitrite be gradually added, the mixture when gently stirred should not form crystals of phellandrene nitrite. It should become semi-solid on being stirred, when cold, with a third or a half of its volume of phosphoric acid of commerce of specific gravity 1.750 (presence of a due proportion of cineol).” The Australian oil fulfils these specifications but the Californian oil does not, and therefore the latter has not been utilized to any great extent. It is this fact that has led us to an examination of the Californian oil, to determine whether there is any difference in the crude oil from the same species of *Eucalyptus* grown in Australia and California, or whether the difference is simply in the method of refining.

Baker and Smith¹ after a very extensive research on the *Eucalyptus* oils of Australia state that “the constituents of the oil of *Eucalyptus globulus* are practically constant wherever the tree is grown; a sample of oil distilled from trees in New South Wales differed in no respect from the product of trees grown in Tasmania or Victoria. The amounts of the constituents may vary but the constituents found are always the same.” Our work on the Californian oil has fully corroborated this statement, since we have found exactly the same constituents in the Californian oil that Baker and Smith found in the Australian oils. The amounts of the constituents vary so greatly, however, as to give the crude oils entirely different properties. This may be seen from the following comparison of the Australian oil and Californian oil.

	CALIFORNIAN OIL	AUSTRALIAN ¹ OIL
D ₂₀	0.9052	0.913
[α] _D ²⁰	14° 42	9° 2
N _D ²⁰	1.46053	?
	Insoluble in 70 per cent alcohol	Soluble in 1.5 volumes of 70 per cent alcohol
Free acid.....	0.73	1.1
Saponification No.....	2.5	1.0
Acetylation No.....	56.0	?

¹“A Research on *Eucalyptus* with Particular Reference to Their Essential Oils,” by Baker and Smith. Published by the Australian Government.

These data were obtained from leaves of the *Eucalyptus globulus* gathered on the University Campus and distilled with steam at a pressure of four pounds; these leaves gave a total yield of oil corresponding to 0.8 per cent of the total weight of the green leaves. Baker and Smith have obtained yields as high as 0.918 per cent; this of course varies somewhat with the season of the year. For the rest of our work, since we wished to make our analysis as valuable as possible commercially, we have used oil obtained from a commercial still in North Berkeley. This oil was obtained from a grove of almost pure Globulus but containing a sprinkling of other varieties and would more fairly represent the average Californian oil than an absolutely pure Globulus oil.

This oil was fractionally distilled and the following table shows the fractions obtained with the constants of each.

Fraction No.	Temperature ° C.	Percentage oil in each	D ₂₀	N _D ²⁰	[α] _D ²⁰
1.....	85-100	3.1	0.866	1.4485	15.25
2.....	100-155	0.8	0.885	1.4606	20.11
3.....	155-165	6.5	0.887	1.4635	19.17
4.....	165-170	35.6	0.896	1.4635	16.00
5.....	170-180	30.0	0.909	1.4645	8.80
6.....	180-190	11.1	0.921	1.4675	3.81
7.....	190 up	12.5	0.980	..	...

From the curves in Fig. II it will be seen that there are at least four constituents in the oil: the first distills between 85° and 155°, the second between 155° and 170°, the third between 170 and 180, and the fourth above 180.

BUTYRIC, ISOVALERIC, AND CAPRONIC ALDEHYDES—
In the fraction boiling below 155° C. the strong irritating odor suggested the presence of aldehydes; on redistillation this part was easily divided into three fractions, the first distilling at 75-85°, the second at 85-100°, and the third at 100-140°. The first of these fractions readily formed an addition product with ammonia which after recrystallization from alcohol melted at 26-27° C. The second one also formed an addition product with ammonia which after recrystallization melted at 52-53°. The first of these fractions was undoubtedly butyric aldehyde, and the second isovaleric aldehyde since the ammonia addition products of these two aldehydes melt at 29°, and 56°, respectively. The third fraction formed no addition products with ammonia but the boiling point and other physical properties indicated capronic aldehyde. A quantitative analysis of the fresh oil, by the sodium

sulfite method of Sadtler and Burgess,¹ showed the total aldehyde content of the oil to be 6 per cent of the total volume.

PINENE—After the removal of the aldehydes the remainder of fractions 1, 2 and 3 was combined with fraction 4 and redistilled. The fraction distilling at 156–157° constituted approximately 20 per cent of the original volume of the oil and the following constants would seem to indicate that it was practically pure pinene.

	Distillate 156–157° C.	PURE PINENE
D ₂₀	0.878	0.8767
[α] _D ²⁰	34°	20–49°
Boiling point.....	156–157°	156–157°
Melting point nitroschloride..	102–103°	102–103°

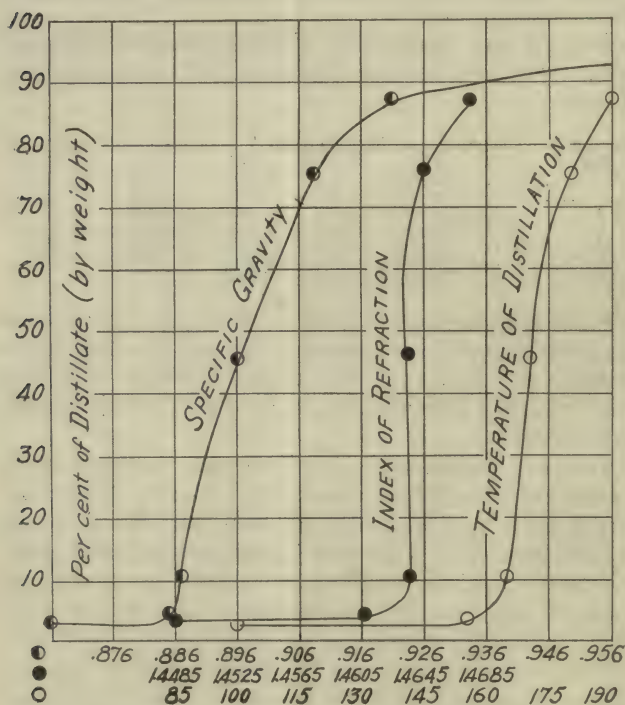


FIG. II—BOILING POINT, SPECIFIC GRAVITY AND INDEX OF REFRACTION CURVES FOR EUCALYPTUS OIL²

A quantitative analysis for pinene was also made on the crude oil as follows: a weighed portion of the oil was fractionated and the fraction boiling at 130–176° retained; 5 cc. of the oil in this fraction were placed in a cassia flask and 50 per cent resorcinol solution was added to take up all the cineol and traces of aldehydes

¹ Burgess, *Analyst*, **29** (1904), 78

² See Burke and Scalione, *THIS JOURNAL*, **6** (1914), 804.

present; after the solution became clear the unabsorbed oil amounted to 21–22 per cent of the whole. This oil had the same properties as the pinene above which had been purified by distillation.

CINEOL—The remainder of the oil from the first fractions was added to fractions 5 and 6, and redistilled; after several fractionations a fraction was obtained which boiled at 176°. A comparison of the constants of this fraction with those of pure cineol shows that this fraction was cineol.

	Distillate 176–177°	PURE CINEOL
Boiling point.....	176–177°	176
D ₂₀	0.930	0.9267
N _D ²⁰	1.457	1.4559
Melting point iodol (after recrystallization) . .	111°	112°

The iodol formed from this distillate after recrystallization melted at 111°. Cineol iodol melts at 112°. A quantitative analysis by the U. S. P. phosphoric acid method gave 47 per cent cineol by volume.

EUDESMOL AND GLOBUOL—The fraction boiling at 190° and up was placed in a shallow dish and allowed to stand for several days until crystals began to separate; these were removed, dried on a porcelain plate, dissolved in alcohol and after adding enough water to produce turbidity allowed to evaporate spontaneously until crystals separated. After another recrystallization soft white flaky crystals were obtained which gave the following constants:

B. p. = 255° M. p. = 80° D₂₀ = 0.988⁷
 $[\alpha]_D^{20} = + 31.21^\circ$ in 12 per cent chloroform solution.

Assuming the formula proposed by Semmler and Tobias,¹ namely, a bicyclic alcohol with an unsaturated grouping, the theoretical molecular index of refraction would be 68.069; the observed molecular index of refraction was 68.001. The acetate prepared by a mixture of acetic anhydride and sodium acetate gave constants identical with those obtained by Tobias and Semmler. This and the general properties of the body seemed to indicate that we were dealing with an alcohol rather than an oxide as suggested by Baker and Smith.

In the higher boiling portions a body corresponding to globuol was separated. Semmler and Tobias consider that this body bears the same relation to eudesmol that borneol bears to isoborneol.

Considering the formulas of the two alcohols to be

¹ Semmler and Tobias, *Ber.*, **43** (2), (1913), 2026.

$C_{15}H_{26}O$ the acetylation number would indicate that the alcohols form 23.22 per cent of the total weight of the oil.

The complete analysis shows that the oil contains the following constituents in the given percentages:

ALDEHYDES (butyric, isovaleric, and capronic).....	6
PINENE.....	21-22
CINCOL.....	47
ALCOHOLS (eudesmol, globuol, with traces of free acids and esters).....	23

In order to determine whether or not the oil from the burrs, which are necessarily gathered in large quantities with the leaves, had any detrimental effect upon the oil as a whole, a quantity of the burrs were

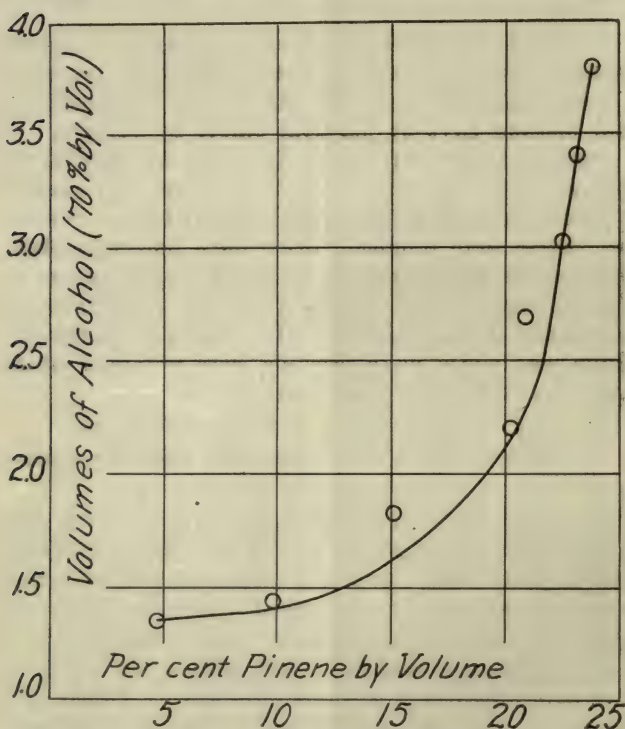


FIG. III.—EFFECT OF PINENE UPON THE SOLUBILITY OF CINCOL

collected, ground and steam-distilled. The yield of oil corresponded to 0.2 per cent of the weight of the burrs. The oil thus obtained was of a lighter specific gravity and had a greater rotation than the leaf oil; it dissolved in 18 volumes of alcohol (70 per cent by weight) while the leaf oil dissolves in 15 volumes. This would seem to indicate that the burrs have a

detrimental effect upon the oil and should be removed as far as possible.

The above analysis would seem to indicate that the chief difference between the Californian and Australian oils is in the exceedingly high content of pinene found in the Californian oil; this would account for the high rotation and low specific gravity of this oil. In order to determine the effect of the pinene upon the solubility in alcohol, pure pinene and cineol were mixed in definite proportions and the solubility in alcohol determined. Fig. III shows the effect of varying concentrations of pinene upon the solubility of cineol. It is readily seen that above certain percentages of pinene the mixture would become practically insoluble in 70 per cent alcohol.

SUMMARY

The above investigation has brought out the following facts:

I—The oil from the *Eucalyptus globulus*, from trees grown in California, has the same constituents as the oil of the Australian trees but in different proportions.

II—The reason why the Californian oil does not fulfil the U. S. P. requirements is probably due in great part to the exceptionally high pinene content.

Since Eucalyptus oil could be easily produced in large quantities in various parts of California and other Pacific coast states, a method of refining the oil so as to bring it up to the U. S. P. specifications would be particularly valuable. The investigation is being continued by the present authors with this idea in view.

CHEMICAL LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY

INVESTIGATIONS ON OIL OF BLACK SAGE

By CHARLES E. BURKE AND CHARLES C. SCALIONE

Received June 22, 1914

In 1912 a bulletin was published by the Bureau of Plant Industry (No. 235), reporting an analysis of the oil of black sage, *Ramona Stachyoides*, of Southern California. This analysis accounted for 62.5 per cent of the oil (camphor, 40 per cent; cineol, 22.5 per cent), but the other constituents were not definitely identified.

Since the black sage grows extensively in Southern California and might become of more or less commercial importance, it seemed to us advisable to continue this work, first with the idea of determining the yields of camphor and cineol at a slightly different season of the year; secondly, to definitely determine the other constituents, and whether or not any of the constituents were present in sufficient quantity to be commercially important.

Through the kindness of Professor C. S. Milliken, Assistant Superintendent of University Extension of Agriculture, several hundred pounds of leaves and twigs which had been simply snipped from the ends of the branches, were forwarded to us from the University Experiment Station at Riverside, in February. These were several weeks in transit and probably suffered some loss from exposure but upon distillation with steam gave a yield of oil corresponding to 0.90 per cent of the weight of material used. This yield is very much higher than that obtained by the Bureau of Plant Industry, who made their distillation about two months later in the season, while the plant was in bloom, and obtained a yield of only 0.75 per cent.

Not only was the yield much greater, but, as might be expected, the physical constants of the oil were quite different from those obtained by the Bureau of Plant Industry, as may be seen from the following comparison:

	Bureau of Plant Industry	Our determination
Specific gravity.....	24° 0.9144	15° 0.8979
" rotation.....	30.2	24.4
Index of refraction.....	1.4682	1.4729
Sol. in 70 per cent alcohol....	Sol. in 1½ to 3½ vol.	Insoluble
Acid No.....	2	2.2
Ester No.....	2.5	1.6
On cooling to -20° C.....	Solid separated.	No solid separated

In order to determine approximately the number of

constituents in the oil and the relative quantities of each, we adopted a method which has been successfully used by L. F. Hawley in "Wood Turpentine" (*Forest Service Bulletin, No. 105*). This method consists in fractionally distilling the oil with a Hempel column and plotting curves as follows:

(a) Temperature of distillation against the per-

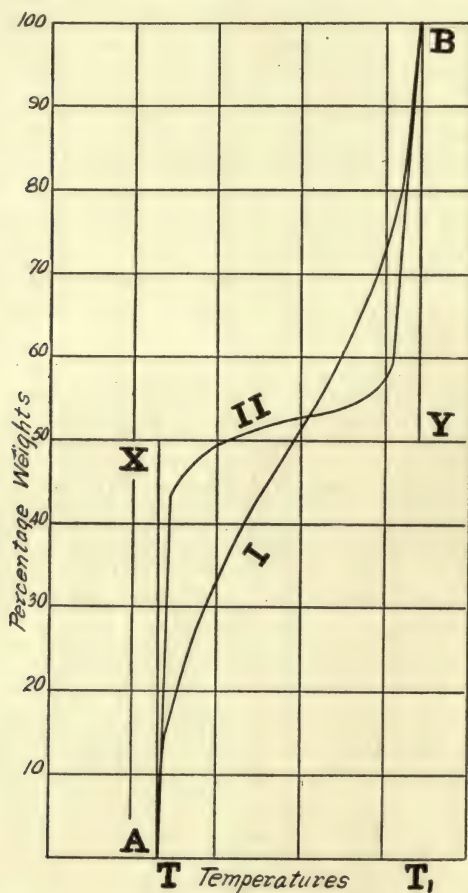


FIG. I—CURVES ILLUSTRATING THE THEORY OF FRACTIONAL DISTILLATION

centage weight which the fraction distilling at that temperature bears to the total.

(b) Specific gravity against percentage weight.

(c) Index of refraction against percentage weight. *Bulletin 105*, Forest Service, gives approximately the following interpretation of these curves.

Suppose we are dealing with two volatile, miscible

components (A) and (B), having boiling points T and T₁, respectively. (A) and (B) are each present to the extent of 50 per cent. If (A) had no effect on (B) then all of (A) would distil over before any of (B) began to come over, and plotting the percentage distillate against boiling points the two vertical lines AX and BY would result. The break in the curve would indicate a change of composition. In an actual distillation this complete separation is never obtained, one component affecting the boiling point of the other. The curve obtained when an ordinary mixture is distilled is represented by Curve I where there is a gradual gradation of the two components. A better separation is obtained by the use of a fractionating column and such separation would be represented by Curve II. In general, approaching the vertical indicates a fairly constant composition, a change towards the horizontal indicates a rapid change of composition. Placing this interpretation upon it, Curve II would represent 45 per cent pure (A), 10 per cent rapidly changing, mixture, and the rest pure (B). In a like manner, the curves obtained from the specific gravity and index of refraction (two of the most constant properties of any oil) should be considered. A rapid change of the specific gravity or index of refraction indicates a rapid change of composition.

TABLE I—FRACTIONATION OF SAGE OIL SHOWING PHYSICAL PROPERTIES OF EACH FRACTION

Fraction	Temperatures ° C.	Per cent distilled over	Specific gravity 15° C.	Specific Rotation 15° C.	Index of refraction 15° C.
1.....	150-160	5.1	0.878	23.9	1.4616
2.....	160-170	1.9	0.892	23.2	1.4619
3.....	170-175	18.6	0.896	22.3	1.4622
4.....	175-180	17.3	0.914	22.3	1.4626
5.....	180-183	6.8	0.926	20.5	1.4633
6.....	183-186	9.3	0.936	21.2	1.4643
7.....	186-195	3.3	0.939	25.9	1.4646
8.....	195-200	6.1	0.930	(a)	(a)
9.....	200-205	18.6	0.940	..	..
10.....	Over 205	12.4	..	..	..

(a) Optical constants not taken, due to formation of solid particles.

Table I shows the fractions we obtained from oil of black sage with the constants for each fraction.

Fraction 1 had a pinene-like odor, Fractions 2-7 had a cineol odor, and the rest of the fractions a camphoraceous odor. A small amount of solid matter began to form in the condenser in Fraction 8 as the 200° mark was reached; this solid matter collected so rapidly in Fraction 9 that the condenser had to be steam-jacketed to carry on further distillation. The specific gravities were determined with a pycnometer, the index of refraction with a Pulfrich refractometer.

From this data the curves in Fig. II were plotted.

An examination of the curves in Fig. II shows at least five components with possible traces of others. The first component is largely separated in Fractions 1 and 2; Fractions 3 to 7 contain at least two constituents and Fractions 8 and 9 two more. The sharp breaks between Fractions 2 and 3 and 7 and 8, particularly noticeable in the specific gravity curves, indicate a sharp separation of the constituents at these points.

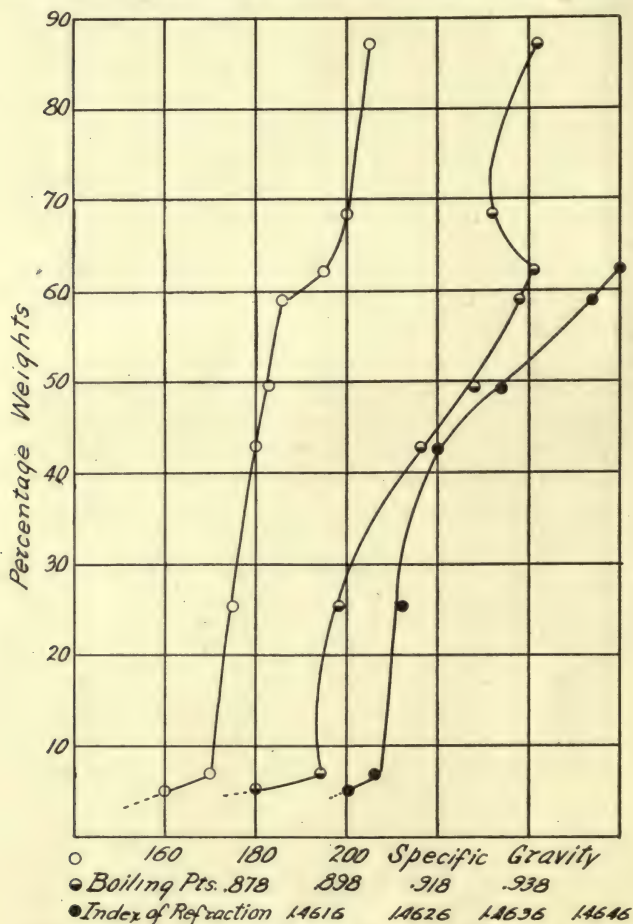


FIG. II—BOILING POINT, SPECIFIC GRAVITY, INDEX OF REFRACTION CURVES FOR SAGE OIL

Fractions 1 and 2 were again redistilled, and with the exception of a small amount of resinous matter, practically all distilled at the boiling point of pinene. This well-cooled, dry oil was saturated with hydrochloric acid and after some time almost completely solidified;

after one recrystallization from alcohol the crystals melted at 130°C . Pinene hydrochloride melts at 131°C . The yield of pinene corresponded to somewhat over 6 per cent of the whole material.

Fractions 3 to 7, after redistillation, were first tested for cineol. To the well-cooled oil, glacial phosphoric acid was slowly added according to Scammel's process.¹ After decanting as much of the oil as possible the magma was placed in a Büchner filter, the filter placed in a centrifuge cup and centrifuged; in this way we were able to get the magma fairly dry without the loss of any of the adhering oil. After decomposition of the phosphate with hot water, a pure oil was obtained, which melted at -1°C . The cineol thus obtained constituted 30 per cent of the whole. The cineol was also identified by the formation of the iodol which, after recrystallization from benzene, melted at $111-112^{\circ}$.

The oil remaining in Fractions 3 to 7 after the removal of the cineol constituted approximately 25 per cent of the whole. This part of the oil was optically inactive and the boiling point and specific gravity indicated either terpinene or dipentene. An attempt was made to form the dipentene tetrabromide, but although crystals separated they were not in sufficient quantity to be identified. On treatment of the oil with Beckmann's reagent² (30 parts water, 6 parts potassium dichromate and 5 parts sulfuric acid), a heavy brown precipitate separated which showed that a considerable portion of the oil consisted of terpinene.

Fractions 8, 9 and 10 were subjected to a freezing mixture of ice and hydrochloric acid, a temperature -20° being attained. Solid crystals amounting to 25 per cent of the total oil separated. After the first sublimation these crystals showed slight optical activity, but after the second sublimation were optically inactive. They melted at 173°C . Pure camphor melts at 175° . The semicarbazone prepared according to the method of Tiemann,³ melted at 232° . The semicarbazone of camphor melts at $236-238^{\circ}$. Undoubtedly these crystals were camphor. After the removal of the camphor there remained approximately 10 to 12 per cent of the original oil. The boiling point was 200°C ., specific gravity 0.917 , and specific rotation about 25.9° . To a solution of the oil in

¹ U. S. Pharm., 1900, p. 313.

² Baeyer, *Ber.*, 27, 815.

³ Tiemann, *Ibid.*, 28, 2191.

petroleum ether, bromine was slowly added according to the method of Wallach¹ for preparing thujone tribromide; crystals were obtained which, after twice recrystallization from ethyl acetate, melted at 123-124°. Thujone tribromide melts at 122°.

A small quantity of oily substance remained from Fractions 8, 9 and 10 after the separation of the camphor and thujone; this was a resinous oil, probably partly formed by polymerization during the process of distillation, and partly consisting of other high boiling terpenes which occurred in quantities too small to be definitely determined.

	Analysis by Bureau of Plant Industry	Our analysis
1 Pinene.....	?	6.0 per cent
2 Cineol.....	22.5 per cent	30.0 per cent
3 Dipentene, terpinene, etc.....	?	25.0 per cent
4 Thujone.....	Trace	8.0 per cent
5 Camphor.....	40.0 per cent	25.0 per cent
6 Resinous material.....	?	5.0 per cent

A comparison of our analysis with that of the Bureau of Plant Industry gives a good indication as to the variation of the constituents of the oil with the season.

At the earlier season at which we obtained our material, the oil contains a considerably higher percentage of the lower boiling constituents, while at the later period a somewhat greater percentage of camphor was found.

CHEMICAL LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY

¹ *Ann.*, **275**, 196; **286**, 129.

THE UTILIZATION OF WOOD WASTE¹

Mark Twain says, "We all talk about the weather, but nothing is done." So it is with wood waste utilization. The lumbermen are interested, of course, but their interest is tempered by weariness and restrained by skepticism. They are open to conviction but would like to see anyone who can convince them. Much the same situation may be found where any of our great industrial wastes are concerned. Those who are responsible for the wastes are so close to them and so long familiar with them that they have come to regard them as the unavoidable accompaniment of the industry. Moreover, plans for waste utilization commonly involve the extension of a business into regions which, however familiar they may be to those doing business in them, are new and strange to the makers of the waste. Conversely, those whose specialized experience might enable them to utilize the waste to advantage, hesitate to incur the large capital expenditure required for establishing an industry dependent upon a raw material over which they have no control and the supply of which may cease at any time, as, for example, when a lumber mill burns down.

These are real and serious obstacles in the way of the utilization of wastes, but the things which anyone can do easily bring no great rewards. The world belongs to those who overcome difficulties and nowhere does it offer richer promise of potential wealth made actual than in the colossal wastes of lumbering.

The figures involved are of astronomical proportions and consequently make little more impression on the mind than the distances of the fixed stars. Let us, however, try once more really to comprehend them. On a total annual cut of 50 billion feet board measure of merchantable lumber, at least 75 billion feet, or about 112,000,000 tons, of wood waste are produced. For every man, woman and child in the country there is therefore annually wasted more than a ton of wood.

The proportion of waste to merchantable lumber varies within wide limits with the kind of wood. In the lumbering of hardwoods, according to Goodman, only 15 per cent of the weight of the standing timber

¹ Address before the 8th Annual Meeting of the American Institute of Chemical Engineers, Baltimore, January 12 to 15, 1916.

appears as finished lumber. Sixty-five to seventy per cent of the original tree is left on the ground and the mill waste amounts to over a cord per 1,000 feet of lumber. Frankforter, who had exceptional opportunities for studying the lumber industry in the middle, northern and western states, reports that in these sections of the country the best equipped and most skilfully operated mills utilize a little less than 40 per cent of the total weight of wood in lumber, lath and shingles. Our own large scale studies on long-leaf yellow pine have proved that under the best operating conditions only 33.42 per cent of the average tree becomes available as lumber, box shooks, lath and shingles; two-thirds is wasted. In the estimate of total annual waste given above an average of 60 per cent of the entire tree was reckoned as waste but in view of the loss on yellow pine, which is our most important timber tree, and the far greater proportionate loss on hardwoods, it is evident that the total annual waste is substantially more than 75 billion feet and may even reach 90 billion feet. Frankforter's estimate of 100 billion feet on a smaller annual cut is undoubtedly too high and not in accordance with his other figures.

Of all our timber trees none lends itself more readily to waste utilization than long-leaf yellow pine. It is cut more largely than any other species and the individual operations are commonly on a great scale which ensures the local concentration of waste in vast amounts. For these reasons and because my associates and I have studied the waste utilization problems presented by this wood more carefully than those of any other species, I shall ignore hardwood distillation, the use of extracted chestnut chips for pulp and paper making, and the occasional use of mill waste from northern conifers in sulfite pulp mills, and ask you to direct your attention solely to the utilization of wood waste from long-leaf yellow pine.

The present annual cut of this species is about 15 billion feet, board measure. The waste is equivalent to 30 billion feet. It may be said at once, without fear of successful contradiction, that the potential profits in this waste are far greater than any actual profits which this branch of the lumber industry can be made to yield from lumber. When this waste is intelligently considered, not as waste but as raw material, it will be seen to afford a basis for building up the greatest group of co-related by-product industries

the world has ever seen. The products of these industries will comprise wood pulp, pulp boards, paper, paper bags, paper twine, turpentine, rosin, pine oil, charcoal, tar, ethyl alcohol, cattle feed, varnishes, ether, and not improbably acetic acid, wood alcohol, acetone and producer gas.

The wood of long-leaf pine is heavy, exceedingly hard, very strong, tough, coarse-grained, compact, durable and very resinous. Its density varies with the height from the ground, the age of the tree and its content of pitch or oleoresin. Sargent gives the average specific gravity as about 0.7, which would seem to be too high: determinations in the Forest Products Laboratory ranged from 0.426 to 0.583. Determinations in our own laboratory gave 0.626 as the specific gravity of round wood averaging 7 inches in diameter. The weight per cubic foot for logging waste we found to be 39.1 lbs. bone-dry. The weight per cord depends greatly upon the shape and size of the pieces. On the dry basis we have found the sawdust to weigh about 1,400 lbs., mill waste with little or no bark 2,340 lbs., with much bark 1,708 lbs.; logging waste in the form of fairly smooth, round logs weighs about 3,260 lbs., and if rough and irregular will average 1,860 lbs., while mature long-leaf pine consisting largely of heartwood will run about 4,300 lbs. to the cord, bone-dry.

We find the ultimate composition of long-leaf pine to be:

Carbon	Hydrogen	Oxygen	Nitrogen	Sulfur	Ash	Total
53.96	7.13	38.65	0.03	0.04	0.16	100 per cent

Our determination of fuel value of average sawmill dust gave 9,240 B. t. u., dry basis. Moisture in green wood averages 34.15 per cent, fresh green stumps average 28.69 per cent, lightwood 13.35 per cent. Stumps about 6 years old carry around 20 per cent of water and kiln-dried lumber contains an average of about 10 per cent. The proportion of bark varies to some extent with the age of the tree and is relatively high. Small, round, Mississippi wood had about 9 per cent, Florida pulp wood over 11 per cent, and Florida trees over 18 inches diameter bore 8.6 per cent of bark by weight. Determinations in our laboratory of the fiber length of long-leaf pine gave a maximum of 7.40 mm., minimum 3.00 mm. with an average of 4.60 mm. as compared with 3 to 3.5 mm. for spruce.

In determining the amount of field waste under careful operation, members of our organization selected

five plots of one acre each as truly representative as possible of the average quality of yellow pine timber. As soon as the timber had been felled the waste between 3 inches and 9 inches was collected, classified and weighed. Much of it was also corded and scaled to determine relations between weight, cord measure and volume. Each stump was measured and its volume and weight computed. Needles from four trees were weighed.

The main points of accumulation of mill waste are the main refuse conveyor and the main dust conveyor. Actual determinations by weight of the amounts of waste carried by these conveyors in very large scale operations were made by members of our staff. The results of these determinations in the field and at the mill enable us to state with a very close approximation to the truth the relative proportions of the initial products of the average yellow pine tree. They have perhaps never been ascertained before within such limits of accuracy or upon so large a scale. The proportions of the several products are as follows:

YIELD FROM ENTIRE TREE		YIELD FROM LOGS	
	Per cent	PERCENTAGE OF WHOLE TREE	
Needles and twigs....	2.25	Red and rotten.....	1.45
Limbs under 3 in.....	2.54	Slabs, edgings and trimmings..	18.07
Cordwood.....	6.42	Sawdust and shavings.....	17.62
Pulpwood.....	4.54	Shingles.....	0.06
Red and rotten.....	6.60	Lath.....	1.39
		Lumber and box shooks.....	31.97
Tops and culls.....	22.35		
Logs, as indicated.....	70.56		70.56
Stumps.....	6.48		
Lightwood.....	0.61		
	100.00		

Comprehensive studies were conducted on these wastes for a period of eight months. The different classes of wastes were carefully and repeatedly analyzed and their content of rosin and turpentine determined. Papers in great variety were made in our experimental paper mill under direction of Mr. V. E. Nunez, and extractions and distillations on the small commercial scale carried out in our Forest Products Department by Dr. L. F. Hawley. The results obtained, together with data from actual commercial practice, point incontestably to the following stupendous totals:

Upon an annual cut of 15 billion feet of yellow pine the lumber industry in our southern states now wastes raw materials sufficient for the concurrent daily production of 40,000 tons of paper, 3,000 tons of rosin, 300,000 gallons of turpentine, and 600,000

gallons of ethyl alcohol, together with fuel sufficient to meet the requirements of all these industries.

Despite the prevailing opinion to the contrary among lumbermen, and even among papermakers, long-leaf yellow pine lends itself admirably to the manufacture of paper. It is too pitchy for the sulfite process but is readily reduced to pulp by the soda process. Much the best results are, however, obtained by the sulfate process, which, when carried out under proper conditions, yields an excellent kraft pulp and paper. The pulp bleaches with some difficulty but may with care be brought to good color without serious impairment of strength. The bleached pulp makes good wood writings and when mixed with pulp from gumwood can be run off into good book papers. A cord and half of round waste makes a ton of paper as against about two cords of spruce costing northern mills from \$9 to \$13 a cord.

The production of ethyl alcohol from yellow pine waste by the Ewen and Tomlinson process has been fully demonstrated as a technical proposition, about 90,000 gallons of high-grade 95 per cent alcohol having been made in the plant of the Standard Alcohol Company at Fullerton, La. The commercial merit of the proposition remains to be demonstrated since this company is now in the hands of the receiver for reasons altogether apart from the technical merit of the process. It may, however, be said that every technical man familiar with the process believes that it is capable of producing 95 per cent alcohol at a cost, including cooerage and all fixed and manufacturing charges, of not more than 20 cents a gallon. Such alcohol is worth to-day about 56 cents a gallon. The yield per cord of sawdust or hogged wood waste containing 50 per cent water is 10 gallons in large scale operation, but yields much higher have been obtained in the laboratory.

The process is based, of course, on the observation of Braconnot in 1819 that cellulose, by treatment with mineral acid, is converted into reducing and fermentable sugars. For nearly 100 years experimenters have endeavored to develop this observation into a commercial process. With the exception of Ewen and Tomlinson all have failed to produce alcohol in commercial quantities. Simonsen in Sweden, in 1889, began a series of brilliant researches which cleared up many points of difficulty and ultimately enabled him to secure very satisfactory results on the large labora-

tory scale. Attempts at commercial operation, however, developed fresh obstacles and led to the abandonment of the process. The well known German chemist, Alexander Classen, patented during the year 1900, 1902 and 1903 various modifications of the general process of producing sugars and alcohol by treatment of comminuted wood with strong acids under heat. Subsequently several plants were built in this country for carrying out one or another of these modifications. In none of them were any substantial amounts of alcohol produced, and the Classen process must be regarded as commercially inoperative. It remained for Ewen and Tomlinson to overcome the fundamental technical difficulties underlying all these attempts and to put forward the first process capable of large scale production of ethyl alcohol from wood.

The Ewen and Tomlinson process is essentially one for producing fermentable sugars. The fermentation of these sugars, once produced, may be carried out and the alcohol recovered in any distillery by the usual methods. It is my belief that the process affords the cheapest known method for producing these sugars and that it therefore is ultimately destined to become an important if not the most important source of industrial alcohol.

In carrying out the process, hogged wood waste, usually containing about 50 per cent of water, is loaded into a rotary digester holding about 7 cords and having a protective and heat-insulating lining. Sulfuric acid in relatively small amount is sprayed upon the wood and then steam is admitted to the digester. The critical temperature is reached as quickly as possible and the reaction is then extremely rapid if not indeed nearly instantaneous. Pains are therefore taken to reduce the pressure immediately and to empty the digester of its contents as promptly as may be. About 25 per cent of the weight of the wood is converted to reducing sugars, not all of which are fermentable.

The cooked material, which is not unlike coarse coffee grounds in appearance, is transferred by conveyors to a diffusion battery or other extraction apparatus in which the sugars are dissolved out. The spent chips go to a continuous press for removal of excess water and are then available as fuel. The extracted juice is neutralized with lime and is then ready for fermentation. The concentrated slop from the stills finds use as cattle feed and is about equivalent to molasses for that purpose.

In a modification patented by me the process may be diverted as a whole to the production of carbohydrate cattle feed. In this modification hydrochloric acid is substituted for sulfuric and subsequently removed so far as is readily possible by heating or blowing air through the mass. The remaining acid is then converted to common salt by the addition of an equivalent amount of sodium carbonate. The juice is extracted and concentrated to the consistency of molasses.

A major economic problem in the South which is bound up with the utilization of wood waste is that of the agricultural development of the cut over lands. It has been demonstrated that by a steam puller yellow pine stumps may be pulled for about 33 cents each. In the trials made the stumps averaged 563 lbs. each, which makes the cost of pulling \$1.16 per ton. Where logging tram roads are still in place, the stumps can be brought to a central plant for treatment at a total cost well within \$2.50 per cord. Treated by solvent extraction they should yield per cord about 6 gallons of turpentine, 2½ gallons of pine oil and 380 lbs. of rosin. The extracted chips, as pointed out by Veitch and Merrill, and amply demonstrated in our experimental paper mill, are available for the manufacture of an excellent grade of kraft paper. Strangely enough, and contrary to our first impression, the charred portions common in such stumps are easily removed in the process and leave the paper clean. The chips are of course also available for alcohol production.

Stumps and the highly resinous wood residues termed lightwood, together with the box slabs from turpented trees, are directly available for treatment by several processes of distillation. The average green wood with 35 per cent moisture contains about 6 per cent rosin but in selected samples of wood the rosin may run higher than 50 per cent. No large quantities are usually to be had, however, containing more than 30 per cent. The composition of the rosin itself is variable but in green wood it runs about 80 per cent rosin and 20 per cent volatile oils; about 80 per cent of the latter are turpentine and the balance pine oil.

Destructive distillation is carried on in retorts operated commonly without temperature control. Average yields under these conditions are:

Light oils	Turpentine	Heavy oil	Tar	Charcoal
6 gallons	6 gallons	16 gallons	48 gallons	35 bushels

Various processes in which the retort temperature is controlled, as in the bath process and the processes of Gautier, Pritchard, and others, have been proposed and several put in operation for a time on either the commercial or large experimental scale. They offer advantages in the way of better yields and quality of turpentine, but these gains are so nearly offset by higher fuel costs and higher maintenance charges that the commercial superiority of these more complicated processes has yet to be demonstrated.

Ten years ago recovery of turpentine from comminuted waste by steam distillation was practiced in many plants, the steam being blown through the mass of chips either upward or downward and carrying the volatile oils with it to the condenser. The decline in the price of turpentine in 1907 and 1908 proved so severe a blow to the industry that it is to-day practically wiped out.

The well known Yaryan process added to the steam distillation method the subsequent step of extracting the chips with a volatile solvent from which the dissolved rosin could later be recovered. Several plants of large capacity were operated successfully until in 1913 the failure of the American Naval Stores Co. caused a heavy drop in the price of rosin which was followed by the failure of the company operating the Yaryan process. A contributory cause may well have been the large losses of solvent incident to the process.

Several methods have been proposed for the recovery of turpentine, pine oil and rosin from yellow pine wastes by the action of water solutions of alkali but none are thought to be operating commercially. They depend upon the fact that the wood is only slightly attacked by the dilute alkali in which the rosin is readily soluble. Nevertheless, some wood is dissolved and the difficulty of separating this so-called "humus" from the rosin has prevented the introduction of processes of this type. The difficulty has been overcome in a simple and ingenious way by Whitaker and Bates, who salt out the rosin soap by increasing, after the initial treatment of the wood, the concentration of the alkaline solution to 3.5 per cent free alkali. The treated chips are then available as a raw material for papermaking.

When the real work of wood waste utilization has

once begun and the attention of chemical engineers and financial men has been drawn more generally to the huge potential values now ignorantly thrown away we may expect the rapid development of these by-product industries and the initiation of many new ones to the great enrichment of the South and in somewhat less degree, that of the Northwest. It would doubtless be too sanguine to expect the directive impulse for this new development to come from the lumbermen themselves. They are too close to the wastes. They are blinded by the sawdust. But if they fail much longer to grasp the opportunity which has been so long beside them they must be content to see others reap the benefits and profits which will come through control of processes, special apparatus, and, above all, of technique.

ARTHUR D. LITTLE

NEW HAMPSHIRE COLLEGE
AGRICULTURAL EXPERIMENT STATION

THE COMPOSITION OF MAPLE SAP

BY F. W. MORSE AND A. H. WOOD



NEW HAMPSHIRE COLLEGE
OF
AGRICULTURE AND THE MECHANIC ARTS
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The Bulletins of this Station are sent to any resident of New Hampshire upon application.

INTRODUCTION.

During the sugar seasons of 1892, 1893, and 1894, Professors Wood and Morse conducted investigations of problems connected with the maple sugar industry. Owing to the brief period in which maple sap flows freely, it has been impossible to make as complete studies in one season as are necessary for accurate knowledge of the subject, and as seasons also vary, scientific conclusions cannot be drawn from the results of two or three years' experiments. Therefore the following pages contain facts which have been observed, but from which few generalities may be deduced.

While the cane, beet, and sorghum have been extensively investigated and many figures recorded, the results of studies of maple sap, thus far published, are few in number. The theories and observations unsupported by details, are, however, very numerous, and the results stated in the succeeding articles will agree with some and contradict others.

The work has been arranged, for convenience, under appropriate heads.

THE COMPOSITION OF MAPLE SAP

F. W. MORSE AND A. H. WOOD

Note. Professor Wood resigned his position on the Station Staff, November 1, 1894.

All work upon maple sap hitherto published, so far as the writer is aware, is recorded in Bulletin 5, Division of Chemistry, United States Department of Agriculture. The results now mentioned are in several cases closely accordant with those in the work mentioned, and are of value on that account.

In the sixth annual report of this station, a detailed description of these investigations has just been published, therefore in this bulletin only the most noteworthy results will be given.

The variation in percentage of sugar has been found to be wide, and some trees have yielded sap which was hardly worth evaporating. Trees with many branches, and exposed to the full effect of the sun, have been found to give the richest saps, and trees with small tops in a thick grove, or much shaded, have given the poorest sap. The amount of sugar in the sap has not depended upon variety of maple, since soft maples have yielded both as high percentages of sugar, and as low as rock maples.

The sap toward the close of the season has shown neither as much sugar, nor as much solids as at the beginning of the season. This change has not taken place at a uniform rate, but instead there have been fluctuations up and down.

VARIATION IN SAP FROM DIFFERENT TREES, MARCH 31, 1892.

Description of Tree.	Per cent. Saccharose.
A. Rock maple, with small top, in a grove.....	1.30
C. Rock maple, with medium top, in a grove beside cart path...	2.30
E. Rock maple, with large, wide top, surrounded by tall, young pines.....	2.80
L. Rock maple, large pasture shade tree.....	5.60
F. Soft maple, with many small branches, in open ground.....	4.00
G. Soft maple, with wide-spread top, surrounded by pines.....	2.50

VARIATIONS IN SAP FROM DIFFERENT TREES. 1893.

Description of tree.	Date.	Saccha- rose.	Total Solids.
2. White maple, situated in a group of maple and hickories; tall, with medium top....	March 22	2.80	3.00
	" 25	2.20	
	" 30	2.90	
	April 13	2.80	
4. Rock maple, near the above tree and similar in size and form. Tapped in two places. Average of results given for each date	March 22	3.40	3.01 2.72
	" 25	3.25	
	" 30	2.88	
	April 1	2.75	
	" 16	2.45	
6. White maple, shade tree by road side, medium size, many branches, well developed.....	March 22	4.90	5.04 3.61 3.37 3.21
	" 25	3.30	
	" 27	3.40	
	" 30	2.90	
7. Rock maple, shade tree by road side, taller and larger than 9, but similarly proportioned.....	April 1	3.00	
	March 22	3.20	
	" 25	2.80	
	" 27	3.10	
	" 30	2.90	
	April 1	2.90	
9. Rock maple, shade tree by road side. About the same size as 10.	March 22	4.00	4.17
	" 25	3.40	
	" 27	4.00	
	" 30	3.40	3.65
	April 13	2.50	
	" 16	2.21	
	" 20	2.35	2.40

The variation in composition of sap from different sides of a tree, has not been found to be wide nor constant. Two trees showed a decidedly higher percentage of sugar from the north side, one tree gave the advantage to the south side, while a fourth tree gave nearly equal percentages to both north and south sides.

COMPOSITION OF SAP FROM DIFFERENT SIDES OF A TREE, 1892 and 1893.

Description of Tree.		Date.	Saccharose.	Total Solids.		
b. Rock maple, in a grove, N. W. side. tall with small top. S. W. side.		1892. Mar. 27	2.25	2.66		
		" 27	1.95	2.12		
2. White maple, in a group of trees, tall with medium top. N. side. S. side.		1893. Mar. 25	1.70			
		" 30	2.60			
		Apr. 13	2.20			
		Mar. 25	2.20			
		" 30	2.90			
		Apr. 13	2.80			
4. Rock maple, near (4.) and similar in size and form. N. side. Average. S. side. Average.		Mar. 22	3.40			
		" 25	3.40			
		" 30	2.75	3.01		
		Apr. 1	2.80			
		" 16	2.51	2.79		
			2.97			
		Mar. 22	3.40			
		" 25	3.10			
		" 30	3.00			
		Apr. 1	2.70	3.01		
" 16	2.39	2.64				
12. Rock maple, in an open field, branches low and wide-spread. N. side. S. side. E. side. W. side.		Apr. 8	2.80			
		" 10	2.95			
		Apr. 8	2.40			
		" 10	2.35			
		Apr. 8	2.60			
		" 10	2.60			
		Apr. 8	2.20			

It has been claimed that the outer wood of the maple yields a richer sap than the inner wood. To test the correctness of this opinion, tree 13 was tapped with two holes as near together as possible; one hole being bored diagonally in order to keep near the bark, and the other bored toward the centre of the tree. Each hole was bored to the same depth on the bit. The result of this experiment was contrary to the above claim. Trees 8

and 10, which had been flowing since March 22 from holes $1\frac{1}{4}$ inches deep, were then bored to a depth of $2\frac{1}{2}$ inches in the same holes on April 9. Samples of sap from each tree were taken before and after the increase in depth of holes. The results were the same as with tree 13, and furthermore, the percentages of sugar continued to remain higher for the balance of the season than they were before the experiment.

On April 20 two more trees, *o* and *p*, were tapped by boring a hole of large diameter to the depth of $1\frac{1}{2}$ inches and then continuing with one of small diameter to an additional depth of 2 inches. The sap was collected from the outer and inner wood by means of a double spout consisting of one tube within another. The results this time agreed with the above claim.

In 1894, two trees, *r* and *s*, were tapped in a similar manner, with the exception that the outer hole was carried to a depth of $2\frac{7}{8}$ inches, and the inner hole $2\frac{7}{8}$ inches further. Again the results agreed with the claim.

VARIATION IN COMPOSITION OF SAP FROM OUTER AND INNER
WOOD. 1893.

DESCRIPTION OF TREE.	Date.	Per cent. Saccha- rose.	Total Solids.
Tree 8. Rock maple shade-tree by road side.	Mar. 22	3.20	3.52
Tapped first on March 22 to a depth of $1\frac{1}{4}$ inches.	" 25	2.90	
On April 9, after collecting the flow of sap, the	" 27	3.20	
hole was extended to the depth of $2\frac{1}{2}$ inches.....	" 30	2.60	
	Apr. 1	2.50	2.86
	" 9	1.65	1.81
Hole bored deeper.....	" 9	2.05	2.34
	" 13	2.06	
	" 16	2.03	2.23
	" 20	1.98	2.18
Tree 10. Rock maple shade-tree by road side.	Mar. 25	4.00	
Tapped in the same manner as 8, and on the	" 27	4.60	4.96
same dates.....	" 30	3.30	4.10
	Apr. 1	3.50	4.37
	" 9	3.45	
Hole bored deeper.....	" 9	3.75	
	" 13	3.73	
	" 16	3.68	4.23
	" 20	3.72	4.16
Tree 13. Large rock maple in an open field.			
Tapped with two holes near together. Hole			
bored diagonally, keeping near the bark.. ..	" 8	2.85	
Hole bored toward centre, 4 inches in depth.....	" 8	3.00	

VARIATION IN COMPOSITION OF SAP FROM OUTER AND INNER
WOOD. 1893-1894.

DESCRIPTION OF TREE.	Date.	Outer Wood.		Inner Wood.	
	1883.	Saccha- rose.	Solids.	Saccha- rose.	Solids.
o. Large rock maple in an open field. Tapped by boring a hole 1" in diameter and 1½" deep, and continuing the tapping with a ½" hole, 2" in depth.....	Apr. 20	2.45	2.53	1.98	2.13
p. Small rock maple growing in a wood. Tapped as above	" 20	2.08	2.25	1.92	1.93
	" 25	2.00	2.17	1.85	
	" 26	2.10	2.30	1.75	1.94
	1894.				
r. Tall maple in a group of trees in open field; 12" in diameter. Tapped with hole 13-16" diameter and 2½" depth, continued by a hole 6-16" diameter and 2½" depth.....	Mar. 13	2.66	2.81	1.90	2.04
	" 17	2.64	2.80	2.36	2.45
	" 22	2.58	2.74	2.05	2.13
	" 30	2.55	2.65	1.86	1.95
s. Rock maple in grove on south slope of a low hill. Diameter about 12".....	Mar. 17	1.99	2.07	1.48	1.54
	" 22	1.86	1.91	1.45	1.56
	" 31	1.93	1.98	1.51	1.59

The variations in richness of sap due to the manner of tapping, have not yet been found sufficiently wide to justify any method which will lessen the flow of sap. By comparing the variations in composition with those of sap-flow given in Bulletin 24, it will be noticed that the methods giving the richest saps yielded the least in quantity.

SOME NOTES ON MAPLE SYRUP AND SUGAR

BY A. H. WOOD AND F. W. MORSE

In 1892 and 1893, some work was done in making maple syrup, but as has been said with regard to sap-flow, the seasons are short and often capricious, necessitating many repetitions of results before laws can be definitely laid down.

The spring of 1892 was a peculiar one, because after the sap had flowed for a period of about two weeks, warm weather ensued which caused the buds to start and stopped the bleeding; and at the end of ten days, the temperature fell, a succession of cold nights occurred, growth was checked and the flow of sap was renewed. The syrups of this second period were scarcely different in composition from those of the first period, except the very earliest.

Experiments in letting sap stand for several days before boiling, filtering sap, and rapid and slow evaporation had no decisive effect on the composition of the syrup.

The syrups from soft maples were somewhat inferior to those from rock maples both in color and flavor.

Delay in boiling sap did not seem to affect the color of the syrup, but injured its flavor. Sap that was kept five days and then boiled gave one of the lightest colored samples produced.

The rapidity of boiling had little influence on the color, samples of syrup from saps that we allowed to slowly simmer away, being as light colored as those from similar saps boiled rapidly.

The lightest colored samples were produced by boiling a quantity of sap until finished, without addition of fresh sap.

One sample produced by boiling about two quarts of sap in a large glass beaker until it was thick syrup, without addition of sap and without skimming, had little more color than the sap from which it had been made. This sap was from covered buckets and was thoroughly strained through cloth before boiling.

Sap filtered through quartz sand produced a syrup in no way superior to the preceding, while one filtered through bone black lost almost entirely the characteristic maple flavor.

Sap mixed with rainwater gave a syrup objectionably dark colored.

To produce a light colored and fine flavored syrup requires that the sap be kept as free as possible from all impurities and thoroughly strained; that it be reduced to syrup with the least manipulations possible, taking care in every part of the process that neither sap nor syrup comes in contact with surfaces that may in any way injure their quality.

COMPOSITION OF MAPLE SYRUP. 1892.

DESCRIPTION OF SAMPLE.	Saccha- rose.	Solids	Reducing Sugars.	Ash.	Purity.
1. First run, March 26.....	69.6	71.0		0.57	98.02
2. " " ".....	70.2	71.5	0.22	0.68	98.17
4. One week later, April 2.....	65.8	68.4	0.23	1.10	96.18
8. " " ".....	41.0	43.9	0.14	0.46	93.40
9. Just before warm period, April 4	66.0	70.3	0.13	1.15	93.87
10. " " ".....	66.4	70.4	0.14		94.32
16. Just after warm period, April 14	62.2	65.8		1.04	94.54
17. " " ".....	60.1	64.9	0.18	1.15	92.62
24. Late run, April 27.....	57.9	62.7	0.27	1.67	92.34
5. White maple, April 2.....	68.9	70.8	0.14	0.60	97.31
21. " " April 16.....	48.2	50.1	0.13	0.60	96.20
20. " " April 18.....	47.8	47.8	0.12	0.59	96.56
7. Run April 1. Boiled at 221° F....	67.5		0.19		
11. " " Evaporated at low					
temperature 3 days, then boiled	56.4		0.09		
13. Same as 11, after standing 4 days	58.1		0.15		
14. Same as 7, after standing 5 days	65.7		0.23		
17. Run April 14. Boiled at 217° F..	60.1		0.18		
18. " " Filtered through					
bone black and then boiled....	63.1		0.25		
19. Run April 14. Filtered through					
quartz sand and then boiled....	56.6		0.15		
22. Run April 14. Boiled at 215° F..	43.6		0.18		

COMPOSITION OF MAPLE SYRUP. 1893.

SYRUP.	Date.	Saccha-rose.	Total Solids.	Reducing Sugars.	Ash.	Specific Gravity.	Purity.
0. White maple.....	Mar. 25	65.4	68.0	0.225	0.407	1.3289	96.18
13. Rock maple.....	" 25	61.8	67.2	0.200	0.670	1.3270	91.96
12. Rock and white..	" 30	58.5	62.8			1.3081	93.15
14. Rock maple. Soured sap.....	" 30	58.2	63.6	0.540		1.3135	91.49
15. Rock maple. Boiled with frequent addition of cold sap.....	Apr. 10	57.2	63.1	0.070	0.720	1.3079	90.65
16. From same trees as 15. Boiled without adding fresh sap.....	" 10	61.9	68.5	0.216		1.3371	90.36

As a preliminary to the investigation of problems connected with the maple sugar industry, several samples of sugar were obtained from various sources and analyzed in order to gain some knowledge of the composition of the article.

COMPOSITION OF MAPLE SUGAR. 1891.

SAMPLE.	Saccha-rose.	Solids.	Reducing Sugars.	Ash.	Purity.
1. Dry sugar.....	93.3	98.3	0.63	0.99	94.9
3. Dry sugar, very light color.....	87.3	95.4	2.69	1.11	91.5
4. Dry sugar, very light color.....	90.5	96.7	0.92	1.19	93.6
5. Dry sugar, very dark color.....	85.2	96.4	4.51	1.16	88.4
6. Dry sugar.....	88.1	95.5	2.76	0.91	92.2
7. Dry sugar.....	91.7	95.6	1.16	0.88	95.9
11. Dry sugar, very light color.....	93.7	98.6			95.0
2. Cake sugar, last run.....	76.8	91.0	7.03	1.23	84.4
8. Cake sugar, soft.....	83.6	88.6		0.92	94.3
9. Cake sugar, medium.....	85.9	90.3		1.04	95.1
10. Cake sugar, hard.....	86.0	92.3		0.88	93.2

The dry sugars contained more solids than cake sugars but the purity co-efficient was as high in one form as in the other. Dark sugars contained less saccharose and more reducing sugars than light sugars and had a much lower purity coefficient.

In 1892 a few samples were prepared under different conditions, and the analytical results are given below.

COMPOSITION OF MAPLE SUGAR. 1892.

SAMPLE.	Finishing Temperature.	Saccharose.	Solids.	Purity.
1. Early run. Very light, soft fine grain.....	230° F.	82.9	84.9	97.6
2. Early run. Light, coarse grain..	235° F.	85.9	88.6	96.9
3. Early run. Clarified. Golden, medium grain.....	240° F.	84.8	88.0	96.3
4. Early run. Clarified and stirred. Finer and lighter than 3.....	240° F.	85.6	88.5	96.7
5. Early run. Clarified and stirred until cold. Fine.....	240° F.	91.5	94.8	96.5
6. Late run. Clarified. Burned slightly; dark.....	240° F.	82.7	88.0	93.9
7. Late run. Clarified and stirred. Burned; lighter than 6.....	240° F.	83.9	89.5	93.7
8. Syrup had fermented and become slightly acid.....		58.1	89.0	65.3

Clarifying the syrup and stirring it while boiling improved the color, and the stirring increased the amount of sugar and solids. Numbers 6 and 7, prepared in the same manner as 3 and 4, but from a later flow of sap, yielded the same percentage of solids, but considerably less sugar and burned slightly. This result is similar to results obtained at the Vermont Experiment Station. (Bulletin 26.)

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Contribution from the Forest Service, Henry S. Graves, Forester.
May 29, 1914.**SUITABILITY OF LONGLEAF PINE FOR PAPER PULP.**

By HENRY E. SURFACE, *Chemical Engineer in Forest Products*, and ROBERT E. COOPER, *Chemist in Forest Products*.

SOUTHERN PINES FOR KRAFT PULP.

The southern pines have not, until within the last few years, been considered suitable for paper pulp. Their resinous nature is the chief drawback in most processes of paper making. The recent development in Europe, especially in Sweden and Norway, of the sulphate process, however, and the superior quality of the product made from resinous woods has turned attention to longleaf and other southern pines as a possible source of pulp in this country. These pines have long, thick-walled fibers, and also high specific gravities, implying large yields per cord, and therefore seem particularly adapted for the manufacture, at low cost, of strong wrapping papers. The waste wood from the lumber industry in the South suggests a source of cheap raw material.

While the sulphate process can be used in the manufacture of bleaching pulps, its principal product is an undercooked, nonbleaching, brown pulp known as "kraft" pulp, the term, a German one, signifying strength. True to its name, this pulp produces a remarkably strong paper, very resistant to wear.

Kraft papers, which may be made by the soda as well as by the sulphate process, are especially adapted for wrapping purposes. Wrapping papers stand third among the paper products of the United States, being exceeded in amount and value only by news and book papers. In 1909 the production of wrapping papers of all kinds aggregated 764,000 short tons, with a value of \$42,296,000.¹ The value of wrapping papers imported in 1912 was \$846,500.² Complete

¹ Tariff Board Report, Pulp and News Print Paper Industry, 1911, p. 21. Senate Doc. 31, 62d Cong., 1st sess.

² Bureau of Foreign and Domestic Commerce, Monthly Summary of Commerce and Finance for December, 1912, p. 744.

statistics for recent importations of kraft paper are not available, but in 1908, three years after its introduction into the United States, the imports amounted to between 10,000 and 12,000 tons.¹ In 1912 the imports of unbleached sulphate pulp from Sweden alone were approximately 21,600 short tons, and from Norway 8,400 short tons.²

Manila wrapping papers, including the better imitation manilas, have generally been considered the strongest and best wearing, but the light-weight kraft papers give the same service as manilas almost twice as heavy. Although strong, light-weight wrapping papers are made in this country from sulphite pulps, the imported kraft papers and papers made from imported kraft pulps have proved too formidable competitors for even the best wholly-domestic product of this kind. The immediate success and largely increasing use of kraft products has brought on the market imitations, colored to resemble the genuine, made from strong sulphite pulp or from such pulp together with ground, steamed-wood pulp. Although some of them are quite strong in the light weights, they are not equal to the genuine in other ways. The opportunity for developing an increased domestic output of kraft products from native woods is apparent.

The above-mentioned conditions led the Forest Service to conduct a series of tests at the Forest Products Laboratory, maintained in cooperation with the University of Wisconsin, Madison, Wis., in order (1) to determine the suitability of the southern pines for paper pulps; (2) to ascertain the effects of varying cooking conditions in the sulphate process of pulp making; (3) to compare the sulphate process with the soda process. Only longleaf pine has so far been used in the tests, of which this bulletin gives the results under such preliminary analyses as have been made at this time.

LUMBER WASTE AVAILABLE FOR PULP MAKING.

The total stand of longleaf pine (privately owned) was estimated by the Bureau of Corporations in 1910 at 232 billion feet board measure, while for all southern pines the amount was placed at 384 billion feet. The lumber cut from these pines in 1910 amounted to 14 billion feet. The sawed lumber represents approximately one-half the volume of the log as it comes to the mill. Bark and sawdust, which are valueless for paper making, constitute a large proportion of the waste, but it is safe to say that 20 per cent of the volume of the log, exclusive of the bark, is lost in slabs, edgings, and trimmings. Tops and defective logs left in the woods and small logs which at present are converted into lumber with little or no profit would furnish a supply of raw material for pulp making even greater than that derived from the mill waste.

¹ Pulp and Paper Investigation Hearings, 1909, Vol. V, p. 3041. House Doc. 1502, 60th Cong., 2d sess.

² From estimates made by the Swedish Wood Pulp Association in 1913 and furnished the Forest Service by Mr. M. Gintzler, New York City.

The waste wood mentioned is not as a rule the clean, clear material to which pulp mills have been accustomed. But when the soda and sulphate processes are employed, the presence of knots, pitch pockets and streaks, and remnants of decayed wood and bark are not very objectionable. The expense of handling and preparing slabs and other irregular sizes and shapes, however, is greater than for round pulp-wood, so the initial cost of such material must be low enough to offset the extra cost incident to its use.

PULP MAKING PROCESSES APPLICABLE TO LONGLEAF PINE.

Four or five mills are at present using southern pine mill waste for the manufacture of wrapping paper and similar products, three of which employ the sulphate process. Several other sulphate mills are either projected or in course of construction. Because of the resinous nature of the wood the preparation of paper pulp from longleaf pine is confined to the soda and sulphate processes, unless special extraction treatments are employed preliminary to cooking.

The soda process consists in digesting suitably prepared wood with caustic soda (NaOH) solution. The cooking results in dissolving the lignin and resin constituents of the wood, and separating the individual fibers from one another. The action depends partly upon the direct solvent and saponifying power of the caustic soda, and partly upon the hydrolysis of the wood in the presence of water at high temperatures, forming organic acid products which unite with the alkali present. Cellulose, of which the fibers are chiefly composed, withstands the cooking action, except under very severe treatment.

The spent cooking liquor, or "black liquor," is separated from the pulp fibers and evaporated; the residue is calcined in a furnace, and the soda compounds are recovered as "black ash," an impure sodium carbonate (Na_2CO_3). This ash is dissolved in water, and the solution is causticized with freshly burned lime; the resulting caustic soda is again used in cooking. The losses of soda occurring in the operations are made up by adding fresh soda ash (commercial sodium carbonate) previous to causticizing.

The sulphate process is similar to the soda process, except that sodium sulphide (Na_2S) is employed as a cooking chemical in addition to the caustic soda. The sodium sulphide is derived from sodium sulphate (Na_2SO_4), which is added during the recovery operations to make up for the losses, and it is from this chemical that the process derives its name. The sodium sulphate is mixed with the black ash and subjected to a high temperature in a "smelter"; this treatment reduces it to sodium sulphide, although the reaction is not complete. The "smelt," containing sodium carbonate, sodium sulphide, and unreduced sodium sulphate, is dissolved in water and the solution is causticized, as in the soda process, with lime, which has, however,

little action on the sulphide and the sulphate. During cooking the organic acids produced react with the sodium sulphide¹ as well as with the caustic soda, so that in calcining both chemicals are recovered as sodium carbonate. If desired, soda ash may be added to the smelt solution before causticizing in order to increase the proportion of caustic soda in the cooking liquors. Some mills have also found it advantageous to mix with the causticized cooking liquors some of the black liquors diverted from the recovery operations.

The soda and sulphate processes can be applied to extracted or steam-distilled chips from which rosin and turpentine have been removed. Turpentine can also be obtained from resinous chips during the cooking operations by condensing the "relief" from the top of the digester. However, the turpentine is very impure, and in the case of the sulphate process contains organic sulphur compounds from which it is separated with great difficulty.

EXPERIMENTAL METHODS.

KINDS OF TESTS.

The tests made by the Forest Service were of two classes: (1) Autoclave tests and (2) semicommercial tests. The autoclave tests comprised several series of cooks made to determine the effects of varying the cooking conditions of the sulphate process. The semicommercial tests include cooks made by the soda as well as by the sulphate process. The semicommercial sulphate cooks employed such cooking conditions as the autoclave tests indicated would give good results, while the tests using the soda process were made with cooking conditions that would give results comparable to those obtained from the sulphate cooks. Because the semicommercial tests show in a more direct manner the possibilities of preparing paper pulp from longleaf pine, they will be discussed before the autoclave tests.

WOOD USED.

The test material consisted of longleaf pine (*Pinus palustris* Mill.) from two localities, Perry County, Miss. (shipment L-3), and Tangipahoa Parish, La. (shipment L-176). A portion of the former, consisting of edgings containing approximately equal amounts of sapwood and heartwood, was used for cooks 176-1, 2, and 3 of the semicommercial soda tests (Table 3), and another similar portion of the same shipment was used for cooks 1 to 65, inclusive, of the autoclave tests. The average bone-dry weight of the wood used in these autoclave tests was 30.4 pounds per cubic foot green volume; the maximum and minimum values were 36.4 and 26.6 pounds, respectively. The wood was fairly free from resin. The remaining cooks employed

¹ In this reaction volatile organic sulphur compounds having extremely disagreeable odors are produced. Unless these odors are eliminated, or held in check by proper means, sulphate pulp mills are highly objectionable except in sparsely populated regions.

two butt logs (15 and 22 inches diameter) of the Louisiana wood, including all of the sapwood and heartwood. These logs were quite resinous, but were free from knots. They had an average bone-dry weight of 35.5 pounds per cubic foot green volume. The maximum and minimum weights were 40.1 and 32.3 pounds, respectively, for the various determinations.

The material was prepared for cooking by removing the bark and sawing the pieces across the grain into sections five-eighths inch thick, which were then split into chips about three-sixteenths to one-fourth inch by 2 to 6 inches across the grain. The chips were screened to remove sawdust, and each lot was thoroughly mixed so as to be uniform throughout.

APPARATUS.

The semicommercial cooks were made in a vertical, stationary digester¹ consisting of a cast-steel cylindrical shell with top and bottom cones, with a capacity of about 62 gallons. The digester was fitted at the top with a "relief" or vent pipe, a pressure gauge, and a thermometer; and at the side with a gauge glass for noting the height of the liquor. The bottom was arranged for "blowing" the contents after cooking. Heat was furnished partly by passing steam directly into the digester at the bottom and partly by two steam coils placed inside the bottom cone. The pressure and temperature were regulated by admitting either more or less steam into the digester and by relieving any excess pressure by means of the top vent.

The autoclave cooks were made in a horizontal rotary autoclave with a capacity of about 2 gallons. This vessel was made of a 6-inch steel pipe with blank flange ends, fitted with trunnions, to one of which was attached a pressure gauge. A screw-joint handhole opening in the side provided for charging. Heat was furnished by Bunsen-burner flames underneath the autoclave, and the pressures were regulated by increasing or decreasing the heat. The autoclave was not relieved during cooking, and no observations of temperatures were made. The cooked pulps were not blown, as in the case of the semicommercial tests, but the cooking vessel was quickly cooled and the contents poured out.

PROCEDURE IN TESTING.

The liquor charges for the sulphate cooks were prepared by mixing caustic soda and sodium sulphide solutions of known composition, as determined by previous analyses, together with water and dry sodium sulphate. The amounts of each constituent were taken in such proportions that when the whole mixture was charged, with the chips,

¹ The apparatus used in the semicommercial cooks is practically the same as that fully illustrated and described in U. S. Department of Agriculture Bulletin No. 80, "Effects of Varying Certain Cooking Conditions in the Productions of Soda Pulp from Aspen," by Henry E. Surface, 1914.

into the digester or autoclave, the amounts of each chemical per pound of chips (bone-dry basis) was in the desired proportion, and the concentration of chemicals in the digester liquor (including the water in the chips) was of the desired degree. For soda cooks the procedure was similar, except that caustic soda was the only chemical to be taken into consideration. The general procedure in conducting the tests was as follows:

The chips to be used for a cook were sampled and weighed. By means of the sample the amount of moisture in the chips and the equivalent bone-dry weight of the charge were determined. The chips, together with the cooking liquors, were then charged into the autoclave or digester, and the vessel closed. After a cook was completed the crude pulp obtained was washed thoroughly, pressed to remove water, shredded, weighed, and sampled for determining its equivalent bone-dry weight. The pulp was then mixed with water and treated in a Hollander-style beating engine¹ with the roll barely touching the bedplate (light brush) until the soft chips in the pulp had become disintegrated into fibers and the wet fibers had a smooth, slippery feel. The beater roll was then pressed hard down on the bedplate (stiff brush), and the beating operation continued until the pulp was suitable for making wrapping paper, as determined by its "feel." The beaten pulp was then screened through the slots (0.012 inch width) of a diaphragm pulp screen. In all cases the screenings obtained were so small in amount that they were disregarded in the yield calculations. The semicommercial pulps were run over a Pusey and Jones 15-inch Fourdrinier paper machine into rolls of dry paper, while the autoclave pulps were made up into sheets on a small hand mold. The papers thus produced contained the experimental pulps alone, without the addition of any other materials.

DETERMINATION OF YIELDS AND PROPERTIES.

The yield of pulp (bone-dry basis) is usually expressed as a percentage of the bone-dry weight of the chip charge, both weights being determined as explained above. When yields per cord are given they are based on a "solid cord" containing 100 cubic feet of clear wood (green volume) having a bone-dry weight of 35.5 pounds per cubic foot;² or 3,550 pounds per cord.

The strengths of the papers from the semicommercial pulps were determined by means of a Mullen paper tester, five "pop tests" being made on double thicknesses of each paper. The value is expressed as a "strength ratio," which is the average of the five test values in pounds per square inch divided by the average sheet thicknesses

¹ A 25-pound Emerson beater was used for the semicommercial tests and a 1-pound Noble and Wood beater for the autoclave tests. Both makes were equipped with steel fly bars and steel bedplate bars.

² This was the average bone-dry weight of the two butt logs of long leaf pine from Louisiana, the material used in the tests for which yields per cord are given.

in ten-thousandths of an inch, and also as a "strength factor," which is the average of the five pop tests divided by the weight per ream of 500 sheets of paper, each measuring 24 by 36 inches. The relative resistance of the papers to wear was determined by crumpling the sheets in the hand, and all other properties mentioned, except strength, were determined by feel or by observation without the aid of instruments.

DEFINITIONS OF TERMS USED.

While the significance of most of the terms used in recording the test data (Tables 1 to 10, inclusive) is either self-evident or sufficiently clear in view of the previous discussion, there are several which may require explanation.

Water in chips.—The amount of moisture is expressed in percentage of water, based on the calculated bone-dry weight of the chips.

All sodium compounds as Na_2O .—This is the sum of the sodium oxide (Na_2O) equivalents of the amounts of the several constituents entering into the chemical charge. "Total Na_2O " has an analagous significance in the soda process.

Sulphidity.—The sulphidity of the liquor charge is the percentage ratio of the Na_2O equivalent of the amount of sodium sulphide (Na_2S) used to the amount of all sodium compounds present expressed as Na_2O .

Causticity.—This has a similar significance with respect to the amount of caustic soda (NaOH) used.

Initial volume of digester liquors.—The digester liquors include the water in the liquor charge, together with the water in the chips and the water condensed from the steam passed into the digester during cooking. This condensation, of course, does not enter into the calculation of the initial volume.

Apparent condensation.—The apparent condensation is the difference between the calculated initial volume of the digester liquors and the observed volume, as read from a water gauge, at the end of the cook. It roughly represents the amount of steam condensing in the digester during cooking, but does not take into account the volume of the pulp and the differences in temperature of the initial and final liquors, nor the steam and liquid lost during relief.

SEMICOMMERCIAL TESTS.

SULPHATE PROCESS.

The object of the semicommercial sulphate cooks was to secure the best quality of pulp with the highest possible yield. The severity of cooking employed depends largely upon the use for which the pulps are intended. If bleaching or easy bleaching pulps, such as are used in book and other white papers, are desired, more severe cooking

treatments are necessary than if the pulps are to be used in natural-color wrapping papers. The present experiments apply more especially to the latter, for which the important properties are strength, toughness, and resistance to wear. The terms mild, medium, and severe cooking, and undercooked, well-cooked, and overcooked pulps used in the following discussion are significant only with respect to the object of the tests.

MILD COOKING TREATMENTS.

The less severe the cooking of a wood the larger will be the yield of crude pulp. However, there is a point at which the pulp will begin to lose its valuable properties for making wrapping papers. For cook 71 the digesting conditions were outlined to give a much undercooked pulp (see Table 1), but the treatment given the wood was even less severe than is indicated by the recorded data, since a portion of the digester liquor was lost through leakage soon after the cook had been started. The crude unbeaten pulp from this cook was full of soft chips, which, while hard enough to resist the action of a stream of water under pressure, could easily be picked apart with the fingers. The paper made from the beaten pulp had a strength factor of 0.50, was moderately tough, and had fair wearing properties. As a wrapping paper it would be considered of medium grade. The yield, 61.2 per cent, or 2,172 pounds per solid cord, was very high, considering the quality of pulp obtained. Pulps produced under less severe cooking conditions had higher yields (see autoclave tests, pp. 14-24), but the quality was not so good, as evidenced by brittleness, lack of strength, and poor wearing properties.

TABLE 1.—Record of semicommercial tests using the sulphate process.

Cook No.	Weight of chips charged (bone-dry basis.)	Water in chips.	Liquor charge.								Initial volume of digester liquors per pound of chips (bone-dry basis).
			Initial concentrations.						Caus-ticity.	Sul-phid-ity.	
			NaOH.	Na ₂ CO ₃ .	Na ₂ S.	SO ₂ com-pounds as Na ₂ SO ₃ .	Na ₂ SO ₄ .	All sodium com-pounds as Na ₂ O.			
<i>Pounds.</i>	<i>Per ct.</i>	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Gallons.</i>	
¹ 71	38.62	34.6	26.5	1.4	13.2	1.8	13.2	38.5	53.3	27.3	0.679
77	38.61	34.7	44.6	2.7	22.3	2.9	22.1	64.9	53.2	27.3	.538
81	23.97	22.7	60.4	3.2	30.0	4.0	30.0	87.6	53.4	27.2	.300
85	23.97	22.7	36.0	1.9	18.0	2.4	18.0	52.3	53.3	27.3	.500
92	23.97	22.6	48.0	2.4	30.0	4.0	30.0	77.5	48.0	30.8	.300
98	23.97	22.6	28.8	1.4	14.4	1.9	14.4	41.8	53.3	27.4	.500
113	25.38	18.2	34.2	1.9	17.1	2.2	18.5	50.4	52.6	27.0	.700
138	25.38	18.2	59.9	3.0	30.0	3.9	30.0	87.0	53.4	27.4	.400
141	25.38	18.2	60.0	3.3	15.0	2.1	30.0	74.4	62.4	16.0	.400
146	25.38	18.2	26.5	1.4	13.2	1.8	13.2	38.5	53.3	27.3	.680
147	26.67	12.5	26.5	1.2	13.2	1.8	13.2	38.4	53.4	27.4	.680
148	26.67	12.5	26.5	1.2	13.2	1.8	13.2	38.4	53.4	27.4	.680

¹ A portion of the digester liquor was lost, due to leaks during the early stages of cooking.

TABLE 1.—Record of semicommercial tests using the sulphate process—Continued.

Cook No.	Chemicals charged per 100 pounds of chips (bone-dry basis).						Duration of cooking.			Maximum cooking temperature.	
	NaOH.	Na ₂ CO ₃ .	Na ₂ S.	SO ₂ compounds as Na ₂ SO ₃ .	Na ₂ SO ₄ .	All sodium compounds as Na ₂ O.	Total.	At zero gauge pressure.	At maximum gauge pressure.		
	Pounds.	Pounds.	Pounds.	Pounds.	Pounds.	Pounds.	Hours.	Hours.	Hours.	° F.	° C.
171	15.0	0.8	7.5	1.0	7.5	21.8	3.0	0.1	2.8	331	166
77	20.0	1.2	10.0	1.3	9.9	29.1	3.0	.2	2.3	331	166
81	15.1	.8	7.5	1.0	7.5	21.9	3.0	.1	2.5	331	166
85	15.0	.8	7.5	1.0	7.5	21.8	3.0	.1	2.5	331	166
92	12.0	.6	7.5	1.0	7.5	19.4	3.0	.25	2.5	331	166
98	12.0	.6	6.0	.8	6.0	17.4	5.0	.1	4.3	331	166
113	20.0	1.1	10.0	1.3	10.8	29.4	3.0	.25	1.0	331	166
138	20.0	1.0	10.0	1.3	10.0	29.0	3.0	.1	2.8	331	166
141	20.0	1.1	5.0	.7	10.0	24.8	3.0	.1	2.5	331	166
146	15.0	.8	7.5	1.0	7.5	21.8	3.0	.2	2.3	338	170
147	15.0	.7	7.5	1.0	7.5	21.8	3.5	.3	2.8	338	170
148	15.0	.7	7.5	1.0	7.5	21.8	3.5	.2	3.0	338	170

Cook No.	Digester pressures per square inch.		Steam pressure per square inch at digester inlet.	Appar-ent con-densa-tion per pound of chips (bone-dry basis).	Yield of crude pulp (bone-dry basis).		Duration of beater treatment.			Strength ratio of paper.	Strength factor of paper.	Ream weight of papers tested.
	Maximum gauge.	Blow-ing.					Total.	At light brush.	At stiff brush.			
	Pounds.	Pounds.	Pounds.	Gallons.	Per	Lbs. per solid cord.	Hours.	Hours.	Hours.			Pounds.
171	90	40	105	10.20	61.2	2,172	3.5	1.5	2.0	0.60	0.50	76
77	90	40	105	.50	45.3	1,609	3.5	2.0	1.5	1.15	.91	31
81	90	50	103	.58	47.9	1,700	5.0	2.5	2.5	1.08	.93	44
85	90	50	108		52.0	1,846	7.0	3.0	4.0	.91	.87	38
92	90	40	110	.49	48.8	1,733	6.5	2.5	4.0	.86	.70	28
98	90	40	108	.50	51.8	1,839	4.5	1.0	3.5	.60	.56	28
113	90	40	100-95	.32	48.6	1,725	6.0	2.0	4.0	.70	.59	37
138	90	40	110		46.1	1,637						
141	90	40	108		44.2	1,569	8.5	4.5	4.0	1.02	.86	36
146	100	40	105		54.9	1,949	9.0	4.0	5.0	.72	.68	45
147	100	40	115	.41	49.1	1,743	6.5	4.0	2.5	.92	.71	37
148	100	40	115	.63	48.4	1,718	8.5	4.0	4.5	1.02	.77	33

(P. L.—138, S. L.—176.)

¹ A portion of the digester liquor was lost, due to leaks during the early stages of cooking.

SEVERE COOKING TREATMENTS.

The effect of more severe cooking treatments, produced mainly by greater initial concentrations and amounts of active cooking chemicals, was evidenced by the thoroughly cooked or overcooked pulps from cooks 77 and 141 (Table 1). The crude pulps were not only free from chips and shives, but also seemed to be soft and fluffy. The papers made from the beaten pulps, however, were of very superior quality with regard to resistance to wear, toughness, and strength, the strength factors being 0.91 and 0.86 for cooks 77 and 141, respectively. Both pulps became slightly hydrated during the beater treatments, which produced a parchmientizing effect and increased the strength and toughness. Either of the papers could be

rubbed or crumpled for a long time without becoming fuzzy, tearing, or showing signs of wear at the place of friction. The papers had also a soft, smooth, greasy, leather-like feel, and were light brown in color, like the imported kraft papers. The yields were rather low for sulphate kraft pulps. For cook 77 the yield was 45.3 per cent, or 1,609 pounds per solid cord, and for cook 141, 44.2 per cent, or 1,569 pounds per solid cord. Under still more severe cooking treatments longleaf pine pulps become very soft and gradually lose their strength and wearing properties. (See autoclave tests, p. 14-24.)

MEDIUM COOKING TREATMENTS.

The above-mentioned cooks show approximately the higher and lower limits of yield in the production of pulps and papers of good quality. However, the better quality of wrapping papers resulted from pulps having the lower yields, and in attempting to secure this better quality, but with higher yields than were obtained for cooks 77 and 141, cooks 85, 98, and 146 were made. For cook 85 the amounts of chemicals and the initial concentrations were decreased from the corresponding conditions for cook 77, while the duration of cooking and the pressure remained practically the same; for cook 98 a further decrease was made in the amounts of chemicals and in the concentrations, but the duration of cooking was increased; for cook 146 the amounts of chemicals and the duration were practically the same as for cook 85, but the concentrations were decreased while the pressure was increased. The cooking conditions, given in full in Table 1, are briefly summarized in Table 2. The resultant papers were in each case of good quality, being tough and resistant to wear, but they were in general not so strong as those from pulps produced under more severe cooking treatments. The strength factors for cooks 85, 98, and 146 were 0.87, 0.56, and 0.68, respectively. There is little doubt, however, that these values could be increased considerably by employing beating and other refining treatments better adapted for these particular pulps than the treatments given them. The yields obtained were quite high, cook 85 yielding 52 per cent, or 1,846 pounds per solid cord; cook 98, 51.8 per cent, or 1,839 pounds per solid cord; and cook 146, 54.9 per cent, or 1,949 pounds per solid cord.

TABLE 2.—Condensed summary of cooking conditions for cooks 77, 85, 98, and 146.

Cook No.	Liquor charge, initial concentrations.		Chemicals per 100 pounds of chips (bone-dry basis).		Duration of cooking.		Maximum gauge pressure per square inch.
	NaOH.	Na ₂ S.	NaOH.	Na ₂ S.	Total.	At maximum gauge pressure.	
	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Pounds.</i>	<i>Pounds.</i>	<i>Hours.</i>	<i>Hours.</i>	<i>Pounds.</i>
77.....	44.6	22.3	20.0	10.0	3.0	2.3	90
85.....	36.0	18.0	15.0	7.5	3.0	2.5	90
98.....	28.8	14.4	12.0	6.0	5.0	4.3	90
146.....	26.5	13.2	15.0	7.5	3.0	2.3	100

All things considered, cooks 147 and 148, which may also be classed with those of medium severity, gave the best results. These two cooks were made under almost duplicate cooking conditions, approximately as follows: Caustic soda and sodium sulphide charged per 100 pounds of chips, 15 and 7.5 pounds, respectively; initial concentration of caustic soda in digester liquor, 26.5 grams per liter; initial volume of digester liquor per pound of chips, 0.68 gallon; total duration of cooking, 3.5 hours, of which 2.8 hours for cook 147 and 3.0 hours for cook 148 were at a maximum gauge pressure of 100 pounds per square inch.

The crude pulps were slightly raw and contained some soft chips, which, however, broke up in the beater. The pulp from cook 148 was hydrated during the beating treatment to such an extent that the paper made from it had a parchment-like appearance, the individual fibers being scarcely distinguishable from each other. This paper had good wearing properties and was very tough, with a strength factor of 0.77. The pulp from cook 147 was not subjected to so long a beating treatment, and the resulting paper was not parchmentized to the same extent as that from cook 148. It had a strength factor of 0.71, however, was very tough, and showed good wearing properties. The yield from cook 148 was 48.4 per cent, or 1,718 pounds per solid cord, and from cook 147, 49.1 per cent, or 1,743 pounds per solid cord.

EFFECTS OF BEATING.

The mechanical treatment given a kraft pulp has as important an influence on the properties of the resulting paper as the cooking treatment itself. A crude pulp which appears to be of little value can be made into strong high-grade paper if the proper beater treatment is employed, while the best pulps can easily be ruined by improper beating. The use of kollergangs or edge runners preliminary to actual beating, or of stone rolls and bedplates in the beaters, and the determination by successive tests of the refining and beating treatments best adapted for a particular pulp undoubtedly would have resulted in papers of much better quality than those obtained. Nevertheless, many of the experimental papers were equal or superior to commercial kraft papers.

The effect of different beater treatments was shown by a single series of tests on some of the crude pulp from cook 71 (Table 1). Separate portions of the pulp were treated in the 1-pound beater for periods of 0.5, 1, 2, 3, and 4 hours with the roll at light brush. The papers resulting from treatments of 2 hours or less were soft and weak, and had poor wearing properties, but for the longer periods the papers were firm and tough, with good wearing properties. Under the 4-hour treatment the fibers became hydrated, and a parchment-like paper resulted. The fibers of longleaf pine when reduced by the sulphate process seem to take up water and to become hydrated very

quickly. For all of the semicommercial tests previously mentioned (Table 1) this effect, indicated by the smooth, greasy feel of the wet paper stock, was obtained with from 2 to 4 hours' beater treatment.

WOOD REQUIRED FOR 1 TON OF PULP.

It has been shown that sulphate kraft pulps of fairly good strength and toughness can be obtained from longleaf pine with yields (bone-dry basis) as high as 61 per cent, or 2,170 pounds per solid cord¹ in case of wood as heavy as that tested. For the production of the best grades of wrapping papers, which equal or excel in quality the imported sulphate kraft papers, the yield of pulp would be approximately 51 per cent, or 1,800 pounds (bone-dry) per solid cord. This is equal to a ton (2,000 pounds) of nominally air-dry pulp.² However, it should be remembered that for wood either lighter or heavier than that on which this calculation is based the amount required per ton of pulp would be correspondingly greater or less, unless the differences in weight were due to resin alone.³

COMPARISON OF THE SODA AND SULPHATE PROCESSES.

Table 3 contains the record of the semicommercial soda tests. The best results in both yield and quality were obtained in the case of cook 152. This cook employed 20 pounds of caustic soda per 100 pounds of wood at an initial concentration of 79.7 grams per liter and 5 hours' cooking at 110 pounds gauge pressure, the total duration being 6 hours. The resulting paper was very strong (strength factor 0.90) and the feel and wearing properties were also exceptionally good for a soda pulp. The yield was 48 per cent, or 1,704 pounds per solid cord.

TABLE 3.—Record of semicommercial tests using the soda process.

Cook No.	Weight of chips charged (bone-dry basis).	Water in chips.	Liquor charge.				Initial volume of digester liquor per pound of chips (bone-dry basis).	Chemicals charged per 100 pounds of chips (bone-dry basis).		
			Initial concentrations.			Causticity.		NaOH.	Na ₂ CO ₃ .	Total. Na ₂ O.
			NaOH.	Na ₂ CO ₃ .	Total. Na ₂ O.					
	Pounds.	Per cent.	Grams per liter.	Grams per liter.	Grams per liter.	Per cent.	Gallons.	Pounds.	Pounds.	Pounds.
102....	23.97	22.6	84.0	3.4	67.1	97.0	0.538	37.7	1.5	30.1
136 ¹	25.37	18.3	59.9	2.3	47.8	97.2	.400	20.0	7.7	16.0
144....	25.38	18.2	90.0	3.4	71.7	97.2	.400	30.0	1.1	23.9
149....	25.89	12.0	90.2	3.2	71.8	97.4	.332	25.0	.9	19.9
150....	25.89	12.0	90.2	3.2	71.8	97.4	.332	25.0	.9	19.9
151....	25.89	12.0	90.2	3.2	71.8	97.4	.332	25.0	.9	19.9
152....	25.89	12.0	79.7	1.8	62.9	98.3	.301	20.0	.5	15.8
176-1 ² ...	41.89	14.6	90.0	2.7	71.3	97.8	.333	25.0	.8	19.8
176-2 ² ...	41.89	14.6	90.0	2.42	71.2	98.0	.266	20.0	.5	15.8
176-3 ² ...	41.89	14.6	90.0	2.42	71.1	98.0	.267	20.0	.5	15.8

¹ Weighing 3,550 pounds; see p. 6.

² Standard moisture content of 10 per cent or 100 pounds air-dry weight equals 90 pounds bone-dry weight.

³ The average specific gravity (oven-dry weight, green volume) of all of the longleaf pine from Louisiana in the shipment from which the two test logs were taken, including bolts cut higher up in the trunks of the same trees and material from several additional trees, was 0.528. (See Forest Service Circular 213, Mechanical Properties of Woods Grown in the United States, 1913, Table 1.) This is equal to a weight per cubic foot of 33 pounds in comparison with the 35.5 pounds obtained for the two butt logs.

TABLE 3.—Record of semicommercial tests using the soda process—Continued.

Cook No.	Duration of cooking.			Maximum cooking temperature.		Digester pressures per square inch.		Steam pressure per square inch at digester inlet.	Apparent condensation per pound of chips (bone-dry basis).
	Total.	At zero gauge pressure.	At maximum gauge pressure.			Maximum gauge.	Blowing.		
	Hours.	Hours.	Hours.	° F.	° C.	Pounds.	Pounds.	Pounds.	Gallons.
102.....	3.0	0.2	2.5	331	166	90	40	115	0.55
136 ¹	6.0	.2	5.3	331	166	90	45	115	.55
144.....	6.0	.2	5.5	338	170	100	40	110	.80
149.....	6.0	.2	5.0	338	170	100	40	122	
150.....	9.3	.3	8.3	307	153	60	40	120	
151.....	4.5	.2	3.8	361	183	140	40	142	1.02
152.....	6.0	.2	5.0	345	174	110	40	125	.55
176-1 ²	7.0	.5	6.0	338	170	100		110	.94
176-2 ²	7.0	.3	6.0	338	170	100		115	.76
176-3 ²	8.0	.3	7.0	338	170	100		110	.91

Cook No.	Causticity of liquor at end of cook.	Efficiency in use of NaOH.	Yield of crude pulp (bone-dry basis).		Duration of beater treatment.			Strength ratio of paper.	Strength factor of paper.	Ream weight of papers tested.
					Total.	At light brush.	At stiff brush.			
	Per cent.	Per cent.	Per cent.	Lbs. per solid cord.	Hours.	Hours.	Hours.			Pounds.
102.....			47.2	1,676	4.0	2	2.0	0.43	0.41	42
136 ¹			50.9	1,808	6.5	2	4.5	.55	.54	60
144.....			39.8	1,413	6.0	6	0.0	1.04	.84	48
149.....	22.7	76.7	44.1	1,566	6.5	2	4.5	1.04	.84	37
150.....	41.6	57.3	52.0	1,846	9.0	5	4.0	.53	.56	43
151.....	17.6	81.9	37.6	1,335	6.0	6	0.0	.91	.80	46
152.....	11.2	88.6	48.0	1,704	8.5	4	4.5	1.05	.90	37
176-1 ²			48.6							
176-2 ²			51.1							
176-3 ²			50.4							

(P. L.—138, S. L.—176; P. L.—164-1.)

¹ Five pounds of sodium chloride (table salt) per 100 pounds of chips were used in addition to the chemicals indicated. However, it will be shown later that sodium chloride has little or no effect. (See p. 19.)

² Shipment L-3a from Mississippi was used as the test material. Data for these three cooks have been published previously in Forest Service unnumbered bulletin, "Paper Pulp from Various Forest Woods," by Henry E. Surface, 1912. Specimens of natural color and bleached pulps accompanied the data.

Cook 150 afforded a yield of 52 per cent, or 1,846 pounds per solid cord, but the quality was not so good as in the case of cook 152, the paper being quite weak (strength factor 0.56) with a correspondingly low resistance to wear. The papers resulting from cooks 144, 149, and 151 were all of very good quality, having high strength ratios and good wearing properties, but the yields were considerably lower than for cook 152.

Soda pulps from longleaf pine tend to be soft and fluffy, even when slightly undercooked, or chippy. Proper beater treatments will remedy this to some extent, but the pulp does not become so well hydrated nor attain the same smooth, greasy feel during beating as the sulphate pulps, and the resultant papers do not show the parchmentized effect so characteristic of the sulphate papers. On the paper machine soda stock runs "free," while sulphate stock runs "slow," provided, of course, both kinds of stock are handled similarly in the beater.

The soda papers were inferior to the sulphate papers in resistance to wear; the latter could be rubbed and crumpled for a long time without showing signs of wear, while the former had a tendency to become fuzzy and tear under similar treatment. Even those sulphate pulps at very high yields had wearing qualities equal to the best soda pulps. There is little doubt that higher yields of good kraft pulp can be obtained with the sulphate process than with the soda process. Sulphate pulps of fairly good quality can be obtained with yields as high as 61 per cent, while the limit for soda pulps is approximately 50 per cent. With higher yields the soda pulps lose strength and toughness and become brittle. A sulphate pulp with a 60 per cent yield can be made into a medium grade of kraft wrapping paper, while a soda pulp having the same yield will produce only a very inferior grade. Considering bursting strength alone, equally strong papers can be made by either process.

The main advantage of the sulphate process over the soda process is that in the former the pulp can be very much undercooked and still produce a fair quality of paper, while a soda pulp must be comparatively well cooked before a good paper can be made from it. Moreover, the best sulphate kraft pulps were obtained with a total duration of cooking of only 3.5 hours, while in the soda tests 6 hours were required to secure the best results.

AUTOCLAVE TESTS.

The autoclave tests, which, as previously explained, preceded the semicommercial tests, were made to determine the effects of varying the cooking conditions in the production of sulphate pulp. The cooking conditions investigated were:

- (1) Amounts of the various cooking chemical employed.
- (2) Cooking pressures or temperatures.
- (3) Durations of cooking.
- (4) Initial concentrations of chemicals in the digester liquors.

Aside from the chemicals normally present in sulphate cooking liquors—that is, caustic soda, sodium sulphide, sodium sulphate, and sodium carbonate, the effects of sodium chloride and sulphur in conjunction with caustic soda were studied. The tests, Tables 4 to 10, inclusive, were made in series, in any of which all cooking conditions except the one under observation were held as nearly constant as possible.

The amounts of sodium carbonate and of SO_2 compounds expressed as Na_2SO_3 in the cooking liquors were in general small and no mention of them is made in the tabulated data. The amounts of sodium sulphate present are indicated only relatively, except in Tables 6 and 10.

EFFECTS OF VARYING AMOUNTS OF CAUSTIC SODA.

The effect of varying amounts of caustic soda on the yield of crude pulp is shown in Table 4. Two series of tests were made, differing in the amounts of sodium sulphate and sodium sulphide employed. In the first series increasing the amounts of caustic soda from 15 to 90 pounds per 100 pounds of wood resulted in a decrease in the yield of from 52 to 27.7 per cent. This decrease, however, was not directly proportional to the increase of caustic soda used, as values of this chemical between 30 and 50 pounds had little effect in varying the yield. For higher and lower values the effect was quite pronounced. In the second series a larger amount of sodium sulphide was used, and consequently the yields were lower for corresponding amounts of caustic soda, but variations in the amounts of this chemical produced similar effects.

TABLE 4.—*Effect of varying amounts of caustic soda (NaOH) on the yield of pulp.*

Weight of chips charged (bone-dry basis).....	pounds..	0.986	to 1.007
Water in chips.....	per cent..	10.2	to 12.6
Initial volume of digester liquors per pound of chips (bone-dry basis).....	gallons..	0.650	to 0.690
Duration of cooking, total.....	hours.....		3.0
Duration of cooking at zero gauge pressure.....	do.....		0.1
Duration of cooking at maximum gauge pressure.....	do.....		2.3
Maximum gauge pressure per square inch.....	pounds..		90
Total duration of beater treatment (at light brush only).....	hours..		0 or 1

FIRST SERIES.

Cook No.	Liquor charge.					Chemicals charged per 100 pounds of chips (bone-dry basis).			Yield of crude pulp (bone-dry basis).
	Initial concentrations.			Causticity.	Sulphidity.	NaOH.	Na ₂ S. ¹	All sodium compounds, asNa ₂ O.	
	NaOH.	Na ₂ S. ¹	All sodium compounds, as Na ₂ O.						
	Grams per liter.	Grams per liter.	Grams per liter.	Per cent.	Per cent.	Pounds.	Pounds.	Pounds.	Per cent.
31	26.3	13.1	38.1	53.6	27.5	15.0	7.5	21.7	52.0
55	52.1	13.0	57.9	69.7	17.9	30.0	7.5	33.4	42.9
56	69.6	13.0	72.0	74.9	14.4	40.0	7.5	41.4	39.6
57	87.0	13.0	85.6	78.7	12.1	50.0	7.5	49.2	42.1
58	104.4	13.0	99.7	81.1	10.4	60.0	7.5	57.3	40.0
59	121.8	13.0	113.6	83.1	9.1	70.0	7.5	65.3	33.3
60	156.6	13.0	139.3	87.1	7.4	90.0	7.5	80.1	27.7

SECOND SERIES.

29	35.4	44.4	84.2	32.6	42.0	19.9	25.0	47.4	42.3
28	55.3	45.9	101.9	42.1	35.8	30.0	24.9	55.2	38.2
33	52.7	43.9	99.5	41.0	35.1	30.0	25.0	56.6	37.0
54	60.8	43.3	104.5	45.1	32.9	35.0	24.9	60.1	39.9
32	70.1	43.9	113.0	48.5	30.9	39.9	25.0	64.4	34.8
27	73.8	45.9	116.7	49.0	31.3	40.0	24.9	63.3	35.0
53	76.6	43.3	117.0	50.8	29.4	44.1	24.9	67.3	38.6
43	88.2	44.3	128.3	53.2	27.4	49.8	25.0	72.5	36.8
26	88.8	44.4	126.8	54.3	27.9	50.0	25.0	71.4	37.6
49	104.2	43.4	139.2	58.0	24.8	60.0	25.0	80.2	31.8
52	121.6	43.2	152.4	61.9	22.6	70.0	24.9	87.8	31.8
51	139.0	43.5	166.6	64.6	20.7	80.0	25.0	95.8	28.1

(P. L.—138, S. L.—36.)

¹ With a few minor exceptions, the same values apply to the Na₂SO₄.

The best quality of pulp was obtained with cook 31, using 15 pounds of caustic soda per 100 pounds of wood. This resulted in a slightly undercooked product, which came from the autoclave in a

form of soft chips. The chips did not break up during the washing operation, but were readily pulped by beater treatment. The pulp was strong, tough, and resistant to wear. When larger amounts of caustic soda were employed the pulp tended to be soft, fuzzy, and less strong, while for smaller amounts it was harsh and brittle. (See cooks 39 and 40, Table 6.) In the second series of tests (Table 4) the conditions were such that all of the pulps were overcooked if considered for kraft papers.

The higher the amount of caustic soda employed, the lighter in color was the pulp. The extremes for the first series of tests were brown in the case of cook 31 and light gray in the case of cook 60. For the second series of tests the color change was less noticeable.

EFFECTS OF VARYING AMOUNTS OF SODIUM SULPHIDE.

The effects of varying the amount of sodium sulphide were shown by three series of tests employing different amounts of caustic soda and of sodium sulphate. The cooking conditions and resultant yields are given in Table 5.

TABLE 5.—*Effect of varying amounts of sodium sulphide (Na_2S) on the yield of pulp.*

Weight of chips charged (bone-dry basis).....	pounds.....	0.996 to 1.043
Water in chips.....	per cent.....	11.0 to 16.0
Initial volume of digester liquors per pound of chips (bone-dry basis).....	gallons.....	0.662 to 0.683
Duration of cooking, total.....	hours.....	3.0
Duration of cooking at zero gauge pressure.....	do.....	0.1
Duration of cooking at maximum gauge pressure.....	do.....	2.0 to 2.5
Maximum gauge pressure per square inch.....	pounds.....	90
Total duration of beater treatment (at light brush only).....	hours.....	0, 1, or 2

FIRST SERIES.¹

Cook No.	Liquor charge.					Chemicals charged per 100 pounds of chips (bone-dry basis).			Yield of crude pulp (bone-dry basis).
	Initial concentrations.			Causticity.	Sulphidity.	NaOH.	Na ₂ S. ²	All sodium compounds, as Na ₂ O.	
	NaOH.	Na ₂ S. ²	All sodium compounds, as Na ₂ O.						
	Grams per liter.	Grams per liter.	Grams per liter.	Per cent.	Per cent.	Pounds.	Pounds.	Pounds.	Per cent.
31	26.3	13.1	38.1	53.6	27.5	15.0	7.5	21.7	52.0
34	26.3	26.3	49.6	41.1	42.2	15.0	15.0	28.3	47.4
35	26.3	43.9	62.4	32.7	56.0	15.0	25.0	35.5	44.5
36	26.3	70.2	88.0	23.2	63.5	15.0	40.0	50.0	39.9
37	26.5	88.3	103.8	19.7	67.5	15.0	50.0	58.9	40.3

SECOND SERIES.¹

133	27.2	1.8	31.1	67.7	4.6	15.0	1.0	17.2	68.9
134	27.2	5.4	34.4	61.3	12.6	15.0	3.0	19.0	67.6
139	27.0	9.0	37.3	56.1	19.2	15.0	5.0	20.7	60.1

THIRD SERIES.¹

129	35.3	3.7	39.4	69.4	7.4	20.0	2.1	22.3	64.3
130	35.9	7.2	43.0	64.8	13.3	20.0	4.0	23.9	53.7
131	36.0	10.8	46.3	60.2	18.5	20.0	6.0	25.7	49.7
132	36.0	14.4	49.6	56.2	23.1	20.0	8.0	27.6	47.7

(P. L.—138.)

¹ The Mississippi wood (shipment L-3b) was used for the first series and the Louisiana wood (shipment L-176) for the second and third series.

² With a few minor exceptions, the Na_2SO_4 amounted to one-half of these values for the first and third series and to two-thirds of these values for the second series.

In the first series of tests, with an increase in the amount of sodium sulphide from 7.5 to 50 pounds per 100 pounds of wood, the yield decreased from 52 to 40.3 per cent. For amounts of 25 pounds or less the pulps were of good quality, being strong with good wearing properties, but for larger amounts the pulps became soft and fuzzy and evidently were overcooked.

In the second series, increasing the amount of sodium sulphide from 1 to 5 pounds per 100 pounds of wood resulted in a decrease in the yields from 68.9 to 60.1 per cent. The largest amount (5 pounds) afforded the best pulp, considering strength and wearing properties; the other pulps were much undercooked and quite brittle.

The third series of tests, using a larger amount of caustic soda (20 pounds), showed the effect of increasing the amount of sodium sulphide from 2.1 to 8 pounds per 100 pounds of wood. Under these conditions, the yield was decreased from 64.3 to 47.7 per cent. The pulp obtained when using 2.1 pounds of sodium sulphide was slightly undercooked and somewhat brittle. The other pulps had fair strength and wearing properties and could be used for making a medium grade of wrapping paper.

As the amount of sodium sulphide was increased, the disagreeable odor arising from the cooking was more noticeable, being much more offensive for cook 37 (50 pounds Na_2S per 100 pounds of wood) than for cook 31 (7.5 pounds Na_2S). Increasing the amount of sodium sulphide resulted in lighter-colored pulps, that from cook 37 being considerably lighter in color than from cook 31.

Sodium sulphide is not so severe in its action on wood as caustic soda. A cook of 8 hours' duration was made with sodium sulphide only, using 40 pounds per 100 pounds of wood and a maximum cooking pressure of 100 pounds per square inch. A yield of 41 per cent was obtained, while a similar cook using caustic soda alone in the proportion of 20 pounds per 100 pounds of wood had a yield of 44.3 per cent. This indicates that caustic soda is almost twice as effective as sodium sulphide in reducing the wood to pulp. The color of the pulp produced when using caustic soda alone was lighter than when using sodium sulphide alone.

EFFECTS OF SODIUM CARBONATE.

Sodium carbonate occurs in the commercial sulphate liquors due to incomplete causticization. That it is of no assistance in reducing longleaf pine was shown by a cook made with 40 pounds of this chemical, 10 pounds of caustic soda, and 5 pounds of sodium sulphide per 100 pounds of wood. The duration of cooking was 7 hours and

the maximum gauge pressure was 100 pounds per square inch. The product came from the autoclave in the form of hard, black chips which were quite "raw" on the inside; the yield was not determined. In comparison with this result, cook 40 (Table 6), using, per 100 pounds of wood, 10 pounds of caustic soda, 5 pounds of sodium sulphide, and 5 pounds of sodium sulphate (the latter being of no assistance in cooking), also afforded a product in the chip form. These chips, however, were soft, and could easily be picked apart with the fingers. Of the 3 hours' total duration for this cook, 2.3 hours were at a maximum pressure of 90 pounds. The yield was 65.7 per cent. While it is hardly safe to base a general conclusion upon a single trial, this test indicates that sodium carbonate, at least when present in considerable quantity, retards or diminishes the effects of the caustic soda and sodium sulphide.

EFFECTS OF SODIUM SULPHATE.

Sodium sulphate is present in the commercial cooking liquors, due to incomplete reduction of the sulphate to sulphide during the smelting operations. Like sodium carbonate, it is of practically no assistance in cooking. A cook of 3 hours' duration and 90 pounds maximum gauge pressure was made, using sodium sulphate in the proportion of 50 pounds per 100 pounds of wood, which yielded 86.3 per cent, while another cook of the same duration and pressure but without any chemicals whatever (that is, using pure water alone) had a yield of 89.1 per cent. Allowing for experimental errors, there was little difference between the results of these two cooks, and in neither case could the product be beaten into pulp.

A cook was also made, using 40 pounds of sodium sulphate, 10 pounds of caustic soda, 5 pounds of sodium carbonate, and 5 pounds of sodium sulphide per 100 pounds of wood; the duration was eight hours and the maximum gauge pressure was 100 pounds per square inch. Only hard black chips were obtained, of no value whatever for pulp. As in the case of the sodium carbonate, there is an indication that sodium sulphate retards the action of the other chemicals. To prove this further tests are necessary.

EFFECTS OF VARYING ALL CHEMICALS IN SAME PROPORTION.

A series of tests was made varying the amounts of all sodium compounds present in sulphate cooking liquors. The several constituents were kept constant in regard to each other in the proportion of 50 parts caustic soda, 25 parts sodium sulphide, and 25 parts sodium sulphate. For convenience the amounts of the different chemicals have been computed to a common basis, and the combined values are expressed as Na_2O (sodium oxide).

The yields shown in Table 6 varied from 65.7 per cent for 14.5 pounds of total Na_2O per 100 pounds of wood to 36.8 per cent for 72.5 pounds of total Na_2O . The conditions indicated for cook 31 afforded the best results with regard to both yield and quality of pulp produced. With the higher yields the pulps were harsh and had less resistance to wear. Nevertheless, wrapping papers of medium grades could be made from these pulps. The pulp from cook 30 was of good quality, with strength and wearing properties equal to that from cook 31, but the yield was not so high. Cooks 43, 26, and 38 were duplicates of each other, and show the accuracy attained in the yield determinations. The pulps from these three cooks were soft and fluffy, and had poor strength and wearing properties, due to overcooking.

TABLE 6.—*Effect of varying amounts of all sodium compounds on the yield of pulp.*

Weight of chips charged (bone-dry basis).....	pounds..	0.996 to 1.007
Water in chips.....	per cent..	10.2 to 11.5
Causticity of liquor charge.....	do.....	53.2 to 54.3
Sulphidity of liquor charge.....	do.....	27.4 to 27.9
Initial volume of digester liquors per pound of chips (bone-dry basis).....	gallons..	0.675 to 0.683
Duration of cooking, total.....	hours.....	3.0
Duration of cooking, at zero gauge pressure.....	do.....	0.1
Duration of cooking, at maximum gauge pressure.....	do.....	2.0 to 2.3
Maximum gauge pressure per square inch.....	pounds.....	90
Total duration of beater treatment (at light brush only).....	hours.....	0, 1, or 2

Cook No.	Liquor charge, initial concentrations.				Chemicals charged per 100 pounds of chips (bone-dry basis).				Yield of crude pulp (bone-dry basis).
	NaOH.	Na_2S .	Na_2SO_4 .	All sodium compounds as Na_2O .	NaOH.	Na_2S .	Na_2SO_4 .	All sodium pounds as Na_2O .	
	Grams. per liter.	Grams. per liter.	Grams. per liter.	Grams. per liter.	Pounds.	Pounds.	Pounds.	Pounds.	Per cent.
40	17.6	8.8		25.5	10.0	5.0	5.0	14.5	65.7
39	21.2	10.6	10.6	30.7	12.0	6.0	6.0	17.4	60.2
31	26.3	13.1	13.1	38.1	15.0	7.5	7.5	21.7	52.0
30	35.6	17.8	17.8	51.3	20.0	10.0	10.0	28.9	47.0
43	88.2	45.3	45.3	128.3	49.8	25.0	25.6	72.5	36.8
26	88.8	44.4	44.4	126.8	50.0	25.0	25.0	71.4	37.6
38	88.2	44.1	44.1	127.6	50.0	25.0	25.0	72.4	36.6

(P. L.—138, S. L.—3b.)

¹ The Na_2O values for cooks 26 and 38 differ mainly because of different amounts of Na_2CO_3 which are not separately recorded in the table.

EFFECTS OF SODIUM CHLORIDE.

A few tests were made to determine whether or not the use of sodium chloride in conjunction with caustic soda would result in firmer and less fuzzy pulps, more resistant to wear, than are ordinarily produced with the soda process. If this were possible a process might be developed to produce pulps similar to those obtained with the sulphate process without the disagreeable odors so characteristic of it. Table 7 shows a comparison between cooks made with caustic soda alone and with caustic soda and sodium chloride. It is not probable that sodium

chloride has an effect on the yield, as is evidenced by the data for cooks 128 and 137. Both cooks employed 20 pounds of caustic soda per 100 pounds of wood, but the former used 5 pounds of sodium chloride in addition. The yields from the two cooks were identical. The use of sodium chloride appeared to improve the qualities of the pulps somewhat, but they were much inferior to sulphate pulps at similar yields. The few advantages attending the use of sodium chloride preclude the possibility of this modification of the soda process being of commercial value.

TABLE 7.—*Effect of sodium chloride (NaCl) used in conjunction with caustic soda (NaOH) on the yield of pulp.*

Weight of chips charged (bone-dry basis).....	pounds..	0.910 to 1.043
Water in chips.....	per cent..	15.1 to 22.0
Causticity of liquor charge (disregarding NaCl).....	do.....	96.0 to 97.2
Duration of cooking at zero gauge pressure.....	hours..	0.1
Maximum gauge pressure per square inch.....	pounds..	90
Total duration of beater treatment (at light brush only).....	hours..	2

Cook No.	Liquor charge, initial concentrations.		Chemicals charged per 100 pounds of chips (bone-dry basis).		Initial volume of digester liquors per pound of chips (bone-dry basis).	Duration of cooking.		Yield of crude pulp (bone-dry basis).
	NaOH.	NaCl. ¹	NaOH.	NaCl. ¹		Total.	At maximum gauge pressure.	
	Grams per liter.	Grams per liter.	Pounds.	Pounds.	Gallons.	Hours.	Hours.	Per cent.
118	44.2	28.6	15.0	10.0	0.420	3.0	2.5	73.9
122	41.1	20.0	20.0	10.0	.600	4.0	3.5	63.5
128	49.2	12.0	20.0	5.0	.500	6.0	5.3	58.9
137	36.3	0	20.0	0	.662	6.0	5.3	58.9
72	35.2	0	20.0	0	.681	3.0	2.3	71.6

(P. L.—138, S. L.—176.)

¹ The values shown represent common table salt and not the pure chemical.

EFFECTS OF SULPHUR.

Cooks using “flowers of sulphur” and caustic soda as the cooking chemicals produced pulps almost identical with those resulting from the sulphate process. The addition of sulphur undoubtedly imparted to the pulps the resistance to wear and strength not obtainable by the soda process alone. These cooks, however, were characterized by the same disagreeable odor as the sulphate cooks, and this modification of the soda process seems to have no particular technical advantage over the sulphate process except in the matter of control of the cooking liquors.

EFFECTS OF VARYING THE PRESSURES OR TEMPERATURES OF COOKING.

In the sulphate process, as in the soda process, the digester pressures represent the pressure of saturated steam, since no other gases are present in sufficient quantity to affect the pressure. This

was determined by actual test. The digester pressures, therefore, correspond to the temperatures of saturated steam; and values of each may be converted into the other by means of standard steam tables.

Table 8 shows the effect on yield of variations of pressure from 40 to 140 pounds per square inch. As the pressures increased, the yields decreased. Cook 45, with a pressure of 40 pounds per square inch, resulted in a product so much undercooked that no pulp could be prepared from it. The yield, of course, was very high. Cook 46, using a pressure of 140 pounds per square inch, resulted in 50 per cent yield. For intermediate pressures the yields were correspondingly higher.

TABLE 8.—*Effect of varying pressures on the yield of pulp.*

Weight of chips charged (bone-dry basis).....	pounds...	1.000 to 1.005
Water in chips.....	per cent...	10.4 to 11.0
Causticity of liquor charge.....	do.....	53.5
Sulphidity of liquor charge.....	do.....	27.4
Initial volume of digester liquors per pound of chips (bone-dry basis).....	gallons...	0.667 to 0.680
Duration of cooking, total.....	hours...	3.0
Duration of cooking at zero gauge pressure.....	do.....	0.1
Duration of cooking at maximum gauge pressure.....	do.....	2.0 to 2.3
Total duration of beater treatment (at light brush only).....	do.....	0 or 1

Cook No.	Liquor charge, initial concentrations.			Chemicals charged per 100 pounds of chips (bone-dry basis).			Maximum gauge pressure.	Yield of crude pulp (bone-dry basis).
	NaOH.	Na ₂ S. ¹	All sodium compounds as Na ₂ O.	NaOH.	Na ₂ S. ¹	All sodium compounds as Na ₂ O.		
	Grams per liter.	Grams per liter.	Grams per liter.	Pounds.	Pounds.	Pounds.	Pounds.	Per cent.
45	21.3	10.6	30.8	12.0	6.0	17.4	40	(²)
42	21.1	10.6	30.6	12.0	6.0	17.4	80	61.3
39	21.2	10.6	30.7	12.0	6.0	17.4	90	60.2
41	21.1	10.6	30.6	12.0	6.0	17.4	120	54.0
46	21.3	10.6	30.8	12.0	6.0	17.4	140	50.0

(P. L.—138, S. L.—3 b.)

¹ The same values apply to the Na₂SO₄ used.² Wood not cooked; no pulp prepared.

Pulps produced with the higher pressures were stronger and had better wearing properties than those resulting from the lower pressures. With lower pressures the pulps became more and more brittle and gradually lost their soft, pliable, leather-like feel. The pulps resulting from the lower pressures were the more brown in color.

The best pressure conditions for these tests seemed to be from 100 to 140 pounds per square inch. If larger amounts of chemicals had been employed, pulps of the same yield and properties would probably have resulted from pressures of 80 to 100 pounds per square inch.

EFFECTS OF VARYING THE DURATIONS OF COOKING.

Since the time from the start of a cook until maximum pressure was obtained in the autoclave was practically constant (varying from 0.5 to 0.7 hour), only the total duration of cooking will be considered. Table 9 shows how the yields were affected for total durations varying from one to nine hours in three series of tests, using high, medium, and low amounts of chemicals. In the case of the first series, employing very high amounts of chemicals, 55.9 per cent of the wood (giving a yield of 44.1 per cent) was dissolved during two hours of cooking, while by cooking for seven hours longer an additional loss of only 12.8 per cent occurred. Cook 124, with a total duration of but one hour, afforded the best pulp and the highest yield for this series. This pulp came from the autoclave in the form of soft chips, and the resultant paper made from the beaten pulp was firm and strong, with good resistance to wear. The other pulps were soft and fuzzy, due to overcooking. As the duration increased, the color of the pulps changed from brown (cook 124) to light gray (cook 78).

TABLE 9.—*Effect of varying durations of cooking on the yield of pulp.*

Weight of chips charged (bone-dry basis).....	pounds..	0.964 to 1.034
Water in chips.....	per cent..	16.0 to 24.4
Causticity of liquor charge.....	do.....	51.6 to 53.3
Sulphidity of liquor charge.....	do.....	26.5 to 27.4
Initial volume of digester liquors per pound of chips (bone-dry basis).....	gallons..	0.667 to 0.680
Duration of cooking at zero gauge pressure.....	hours.....	0.1
Maximum gauge pressure per square inch.....	pounds.....	90
Total duration of beater treatment (at light brush only).....	hours.....	1.5 or 2

FIRST SERIES.

Cook No.	Liquor charge, initial concentrations.			Chemicals charged per 100 pounds of chips (bone-dry basis).			Duration of cooking.		Yield of crude pulp (bone-dry basis).
	NaOH.	Na ₂ S. ¹	All sodium compounds as Na ₂ O.	NaOH.	Na ₂ S. ¹	All sodium compounds as Na ₂ O.	Total.	At maximum gauge pressure.	
	Grams per liter.	Grams per liter.	Grams per liter.	Pounds.	Pounds.	Pounds.	Hours.	Hours.	Per cent.
124	71.9	36.0	104.7	40.0	20.0	58.3	1.0	0.5	57.4
125	71.9	36.0	104.7	40.0	20.0	58.3	2.0	1.5	44.1
80	72.0	36.0	104.7	40.0	20.0	58.2	5.0	4.3	37.8
78	72.0	36.0	108.1	40.0	20.0	60.1	9.0	8.3	31.3

SECOND SERIES.

123	44.9	22.5	65.4	25.0	12.5	36.4	1.0	0.5	68.6
126	44.9	22.5	65.4	25.0	12.5	36.4	2.0	1.5	48.5
84	45.0	22.7	65.6	25.0	12.6	36.5	5.0	4.3	45.0
83	45.0	22.7	65.6	25.0	12.6	36.5	9.0	8.3	38.2

THIRD SERIES.

86	21.6	10.8	31.4	12.0	6.0	17.4	1.0	0.3	80.9
127	21.6	10.8	31.6	12.0	6.0	17.6	2.0	1.5	66.7
88	21.2	10.6	30.8	12.0	6.0	17.4	5.0	4.3	59.0
87	21.6	10.8	31.4	12.0	6.0	17.4	9.0	8.3	57.0

¹ The same values apply to the Na₂SO₄ used.

In the second series, when medium amounts of chemicals were used, prolonging the durations of cooking likewise resulted in decreasing the yields. The yield for cook 123, with a total duration of one hour, was 68.6 per cent; and cook 83, with a total duration of 9 hours, had a yield of 38.2 per cent. With a 2-hours' duration the amount of the wood dissolved was 51.5 per cent (48.5 per cent yield), while with a 7-hours' longer cooking period the loss was only 10.3 per cent additional. The best kraft pulps were obtained from cooks 126 and 84, with total durations of 2 and 5 hours, respectively. The resultant papers were firm and strong, and resistant to wear. Cook 123, using a duration of 1 hour, resulted in a weak, brittle, and undercooked pulp, while the pulp from cook 83, which had a duration of 9 hours, was soft, fluffy, and evidently overcooked.

The same general effects resulted from varying the durations in the third series of tests in which comparatively low amounts of chemicals were employed. In this case, however, the best pulps were produced with the longer durations, 5 hours for cook 88 and 9 hours for cook 87. The tests employing shorter durations resulted in weak and brittle pulps, due to undercooking. The pulp from cook 88 was slightly inferior to that from cook 87, but both would be considered of fair quality for making kraft wrapping paper.

The results from the three series of tests indicate that cooks employing high amounts of chemicals and very short durations will afford pulps of a quality and yield similar to those obtained when using medium amounts of chemicals and medium durations and to those resulting from the use of low amounts of chemicals and comparatively long durations. It is evident, however, that much more careful control of the operations must be exercised in order to obtain consistent results when high amounts of chemicals are employed.

EFFECTS OF VARYING THE INITIAL CONCENTRATIONS.

In each of two series of tests varying the initial concentrations of chemicals in the liquor charge the amounts of chemicals per 100 pounds of wood were held constant as follows: 15 pounds of caustic soda, 7.5 pounds of sodium sulphide, and 7.5 pounds of sodium sulphate for the first series, and 12 pounds of caustic soda, 6 pounds of sodium sulphide, and 6 pounds of sodium sulphate, for the second series. Since the amounts of chemicals were held constant, and the concentrations varied, the initial volumes of digester liquors per pound of chips also varied accordingly. Table 10 shows the effect of the varying concentrations on the yield of pulp.

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TABLE 10.—*Effect of varying initial concentrations on the yield of pulp.*

Weight of chips charged (bone-dry basis).....	pounds..	0.964 to 1.031
Water in chips.....	per cent..	16.4 to 24.4
Causticity of liquor charge.....	do.....	52.8 to 53.6
Sulphidity of liquor charge.....	do.....	27.2 to 27.5
Duration of cooking, total.....	hours..	3.0
Duration of cooking, at zero gauge pressure.....	do.....	0.1
Duration of cooking at maximum gauge pressure.....	do.....	2.0 to 2.5
Maximum gauge pressure per square inch.....	pounds..	90
Total duration of beater treatment (at light brush only).....	hours..	2
Chemicals charged per 100 pounds of chips (bone-dry basis):		
NaOH.....	pounds..	15.0
Na ₂ S.....	do.....	7.5
Na ₂ SO ₄	do.....	7.5
All sodium compounds as Na ₂ O.....	do.....	21.8
		First series. Second series.

FIRST SERIES.

Cook No.	Liquor charge, initial concentrations.				Initial volume of digester liquors per pound of chips (bone-dry basis).	Yield of crude pulp (bone-dry basis).
	NaOH.	Na ₂ S.	Na ₂ SO ₄ .	All sodium compounds as Na ₂ O.		
	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Grams per liter.</i>	<i>Gallons.</i>	<i>Per cent.</i>
89	60.0	30.0	30.0	87.2	0.300	47.9
90	45.0	22.5	22.5	65.1	.400	53.3
91	36.0	18.0	18.0	52.1	.500	55.2
93	30.0	15.0	15.0	43.6	.600	58.6
94	25.7	12.9	12.9	37.4	.700	61.3
95	22.5	11.2	11.2	32.7	.800	64.4
96	20.0	10.0	10.0	29.0	.900	66.4
97	18.0	9.0	9.0	26.1	1.000	66.9

SECOND SERIES.

112	72.0	36.0	36.0	104.9	0.200	51.0
100	49.7	24.8	24.8	72.0	.290	51.1
101	36.0	18.0	18.0	52.2	.400	52.3
105	28.8	14.6	14.4	42.2	.500	56.0
114	24.0	12.0	12.0	35.0	.600	62.6
106	20.6	10.3	10.3	30.0	.700	60.6
107	18.0	9.0	9.0	26.2	.800	66.0
108	16.0	8.0	8.0	23.3	.900	67.4
115	14.4	7.2	7.2	21.0	1.000	67.3
110	12.0	6.0	6.0	17.5	1.200	67.8
111	10.3	5.1	5.1	15.0	1.400	67.4

(P. L.—133, S. L.—176.)

When the concentration of all sodium chemicals expressed as Na₂O was varied from 26.1 to 87.2 grams per liter (first series of tests) the resultant yield decreased from 66.9 to 47.9 per cent. The best results, considering both yield and quality of pulps, were obtained from cooks 91 and 93, using Na₂O concentrations of 52.1 and 43.6 grams per liter, respectively. Pulps produced from cooks having lower concentrations were brittle and lacked strength and wearing properties. In the second series of tests, using somewhat smaller amounts of chemicals, the higher concentrations afforded the better results. The best pulp with regard to strength and wearing properties was that obtained from cook 112, using a Na₂O concentration of 104.9 grams per liter. The pulps obtained when using a concentration of 35 grams per liter or less were quite brittle, and had little strength and poor wearing properties.

SUMMARY OF CONCLUSIONS FROM THE AUTOCLAVE TESTS.

(1) The effective cooking chemicals in sulphate cooking liquors are caustic soda and sodium sulphide, the former being the more drastic in its action. Sodium sulphate and sodium carbonate, which unavoidably occur in the commercial liquors, are of no assistance in cooking, at least so far as the wood of longleaf pine is concerned.

(2) Increases in the amounts of either caustic soda or sodium sulphide, or both, result in more thorough cooking. The same effect may be obtained by increasing either the cooking pressure, the duration of cooking, or the initial concentrations of the chemicals in the cooking liquors.

(3) More thorough cooking is evidenced by decreased yields and by lighter colored pulps until a condition of very thorough cooking is reached, after which the color of the pulp is not affected.

(4) The best, or well-cooked, sulphate kraft pulps will have good strength and wearing properties, will be light brown in color, and will have a smooth, firm, leather-like feel when properly beaten. Undercooked pulps are characterized by a darker brown color, brittleness, lack of strength, and poor wearing properties. Overcooked pulps are light gray in color and may have good strength and wearing properties when properly beaten, but the yield will be low. Pulps much overcooked, in addition to being light gray in color, will be soft and fluffy, with little strength.

(5) With each different combination of the cooking conditions there is a definite minimum amount of sodium sulphide which must be used in conjunction with the caustic soda present to impart to the product the high strength and good wearing properties characteristic of properly cooked sulphate kraft pulps.

(6) The use of sodium chloride in conjunction with caustic soda improves the quality of the pulp to a slight extent only. The similar use of sulphur results in pulps having properties practically the same as those of sulphate pulps.

(7) As the proportion of sodium sulphide in the digester charge is increased, the disagreeable odor produced in the cooking operations becomes more pronounced.

PRACTICAL SIGNIFICANCE OF THE EXPERIMENTS.

While the present experiments are not complete, they show conclusively (1) that longleaf pine is well adapted for the manufacture of natural-color kraft pulps and papers; (2) that the sulphate process of pulp making applied to this wood affords products of better quality and of higher yields than the soda process; (3) that kraft papers can be made from longleaf pine equal or superior in quality to the imported and domestic kraft papers now on the market; and (4)

that the high gravity of the wood and the resultant high yield of pulp per cord give longleaf pine an advantage possessed by few, if any, other commercially important woods suitable for pulp making.

The autoclave tests indicate that there should be a certain combination of values for the variable cooking conditions which will result in the most economical method of operation. However, other factors than the variables thus far investigated must be taken into consideration in determining what this combination is. For example, the proper degree to which a pulp must be cooked will depend partly upon the cost of the beater treatment. With cheap power for beating, the pulp need not be so severely cooked as when the cost of power is high. The best concentrations and proportions of chemicals in the digester liquors will likewise depend upon the efficiency of the recovery system and the method of operating it.





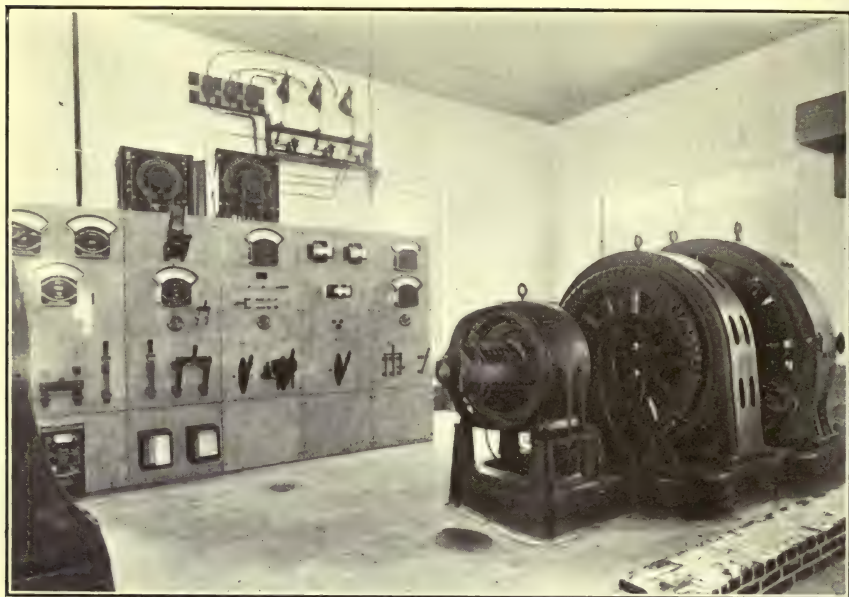


FIG. 1.—MOTOR GENERATOR SET AND SWITCHBOARD.



FIG. 2.—GRINDER AND WET-MACHINE ROOM.

Issued June 11, 1912.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE.

HENRY S. GRAVES, Forester.

FOREST PRODUCTS LABORATORY SERIES.

EXPERIMENTS WITH
JACK PINE AND HEMLOCK FOR
MECHANICAL PULP.

BY

J. H. THICKENS,

Chemical Engineer in Forest Products.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1912.

LETTER OF TRANSMITTAL.

UNITED STATES DEPARTMENT OF AGRICULTURE,

FOREST SERVICE,

Washington, D. C., December 13, 1911.

SIR: I have the honor to transmit herewith a manuscript entitled "Experiments with Jack Pine and Hemlock for Mechanical Pulp," by J. H. Thickens, chemical engineer in forest products, and to recommend its publication.

Respectfully,

HENRY S. GRAVES,

Forester.

Hon. JAMES WILSON,

Secretary of Agriculture.

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EXPERIMENTS WITH JACK PINE AND HEMLOCK FOR MECHANICAL PULP.

NEED OF A SUBSTITUTE FOR SPRUCE PULPWOOD.

Few well-established industries have expanded as rapidly as has the pulp and paper industry. In less than a decade the amount of raw material used annually has more than doubled. During 1900¹ there were consumed in the United States 1,986,310 cords of pulpwood. The ground-wood process used 598,229 cords of domestic spruce, 120,820 cords of imported spruce, and 67,791 cords of other woods, such as hemlock, jack pine, poplar, and balsam, or a total of 786,840 cords. During 1909² the amount of wood used in all processes was 4,001,607 cords, the ground-wood process using a total of 1,246,121 cords, which consisted of 806,282 cords of domestic spruce, 317,289 cords of imported spruce, and 122,550 cords of other miscellaneous woods.

Thus the increase in the total amount of pulpwood used during this period was 101 per cent, while the amount of pulpwood of all kinds used for ground wood increased 58½ per cent. The domestic spruce consumption for this purpose increased 35 per cent and the consumption of miscellaneous woods 80.5 per cent. But the largest increase was in the use of imported spruce, the consumption of which increased 162 per cent.

The price of spruce has increased at a very rapid rate. In 1900 the average cost of spruce used in all processes in the United States was \$4.83 per cord for domestic spruce and \$6.50 for imported, while in 1909 the average price of domestic spruce was \$9.32 and of imported \$11.34 per cord.

This increase has been reflected in the cost of ground-wood pulp. The manufacturing cost of pulp, as determined by the Tariff Board,³ increased from \$10.84 per ton in 1900 to \$16.58 in 1909, 93 per cent of this increase being accounted for by the greater cost of the wood used. Manifestly, therefore, it is almost essential, if the ground-wood

¹ Twelfth Census of the United States.

² "Pulpwood consumption, 1909," Bureau of the Census.

³ Report by the Tariff Board relative to pulp and news print paper industry, Senate Document No. 31, Sixty-second Congress, first session.

industry is to continue, that substitutes be found for spruce pulp, especially in the manufacture of news, wrapping, and other of the cheaper grades of paper. To determine whether there are not other domestic species which will produce a commercial grade of ground wood suitable for the purpose, etc., the Forest Service, in cooperation with the American Pulp and Paper Association, began an extensive series of tests on several of the woods which occur in large quantities in the United States, particularly in the Lake States. The woods which have been tried up to the present are hemlock and jack pine, together with a small amount of spruce, for the purpose of comparison.

The experiments were conducted at Wausau, Wis., under the general supervision of the director and assistant director of the Forest Products Laboratory, and an advisory committee of the American Pulp and Paper Association, composed of Messrs. G. F. Steele, chairman Nekoosa-Edwards Paper Co.; W. G. McNaughton, secretary Nekoosa-Edwards Paper Co.; D. C. Everest, Marathon Paper Mills Co.; W. L. Edmonds, Wausau Paper Mills Co.; A. M. Pride, Tomahawk Paper Co.; and Wm. Eibel, Rhinelander Paper Co.

RESULTS OF EXPERIMENTS.

Not only have very promising sheets of pulp been obtained from both the hemlock and jack pine, but paper has been made from them on commercial machines, operating at high speed, and under all other conditions of actual commercial practice, which has the strength, finish, and appearance of standard news paper. The production per grinder, the horsepower consumption per ton, and the yield per cord approximate the averages which obtain in the grinding of spruce. Again, pulps composed of mixtures of hemlock, spruce, and jack pine in different proportions have been obtained, which compare very favorably with the ordinary spruce ground wood.

Hemlock ground wood has a decided reddish tinge, though this is not very noticeable, even in an all-hemlock sheet of news paper. Jack pine pulp is also slightly off in color, but is not nearly as dark as hemlock pulp. Careful study by experts should make it possible to bring the color of the paper produced from these pulps more nearly to the usual white. As it is, the sheets of news paper which have been secured are only slightly off color, though they are the result in each case of but a single attempt to secure the standard degree of whiteness.

Since the experiments on hemlock have brought out a number of points in favor of the grinding of that wood, two paper-mill companies have signified their intention of using it in their cheaper grades of paper. One of these mills has already begun to do so, and is well satisfied with the pulp obtained.

WHY JACK PINE AND HEMLOCK HAVE NOT BEEN USED.

There is much doubt as to exactly why the pulp industry has neglected to use hemlock and jack pine for the cheaper grades of paper. It seems to be the general impression that hemlock grinds so fine and short that there is a great loss in conversion. It has been said that the yield obtained is in many instances only three-fifths of that from an equal amount of spruce. This loss in grinding hemlock has not been in evidence during the tests.

The pitch in jack pine is undoubtedly responsible for the lack of attention paid to that wood. This, however, can be removed by steaming or soaking, and such treatments will be taken up in future experiments.

In all the experiments the yields secured from the different woods were in direct proportion to their bone-dry weight per cubic foot. It is therefore to be expected that the yields from jack pine and hemlock will be less per unit of volume than those from spruce, since the two first woods are considerably lighter in weight. On the basis of weight, however, there appears to be relatively little more loss in converting hemlock or jack pine into pulp than in converting spruce.

The fiber obtained from the ground hemlock and jack pine has been considered unsatisfactory on account of its shortness. Yet it has been found long enough for use in cheap papers.

One who is accustomed to handling spruce ground wood will not be favorably impressed with the appearance of either hemlock or jack pine pulp. This is particularly true of the hemlock sheet. Both pulps are somewhat softer in texture than spruce, and, altogether, are not as pleasing in appearance as the present commercial product.

Another point which may account for the lack of attention paid to hemlock and jack pine is the care which must be exercised in grinding them. It is possible to obtain a grade of pulp from spruce which is suitable for most purposes without using a great deal of care in the preparation of the surface of the pulp stones. In the grinding of jack pine and hemlock, especially hemlock, on the other hand, great care must be exercised in bringing the stone to the correct degree of sharpness, since these woods will grind to powder if the surface is as sharp as the one ordinarily employed in grinding spruce.

Yet notwithstanding certain shortcomings the fact remains that it is possible to obtain hemlock and jack pine pulps commercially which are suitable for the cheaper grades of paper.

EQUIPMENT USED IN THE EXPERIMENTS.**ELECTRICAL EQUIPMENT.**

To study accurately the fundamental variables of grinding it was necessary to install an elaborate electrical drive and apparatus for control and manipulation. There has also been provided a system of recording instruments for the determination of speed, pressure, and load fluctuation. The electrical apparatus consists of a motor-generator set and a direct-current variable-speed motor. The variable-speed motor, direct connected to the pulp grinder, is rated at 225 horsepower at 100 revolutions per minute and 500 horsepower at 300 revolutions per minute, with a 50 per cent overload capacity at all speeds. However, it is possible to obtain about 25 per cent more capacity than the rating. Variation in speed is obtained by variation of voltage applied to the motor armature, and by means of a rheostat in the field circuit of the generator it is possible to maintain this voltage at any point desired, thus giving a very constant speed.

The efficiency of the grinder motor has been determined for all values of speed and load throughout the range of usage. Consequently, the power applied to the grinder at any value of peripheral speed or at any pressure on the cylinders can be calculated.

Individual motor drives have also been installed for the various other pieces of pulp-making machinery and their auxiliary apparatus. The machines for wood preparation and the wet-machine vacuum pump are the only ones which are not direct connected to individual motors. In several cases variable-speed motors have been installed to permit adjustment of speed to the most effective value.

PULP MACHINERY AND AUXILIARY EQUIPMENT.

The pulp-making machinery, machines for wood preparation, and the auxiliary pieces of apparatus are all of standard commercial types and were loaned by the following manufacturers or others interested in the work of the laboratory:

Grinder.....	Friction Pulley and Machine Works.
Wet machine.....	Improved Paper Machinery Co.
Flat screen.....	Harmon Machine Co.
Ruth centrifugal screen.....	H. L. Orrman & Co.
5 by 8 inch triplex pump.....	Goulds Manufacturing Co.
4 by 6 inch triplex pump.....	Do.
5-inch centrifugal pump.....	Do.
4-inch centrifugal pump.....	Do.
Storage tank.....	Valley Iron Works.
Barker.....	Green Bay Foundry and Machine Works.



FIG. 1.—NATURAL SURFACE OF STONE. (JACK PINE RUN No. 2.)

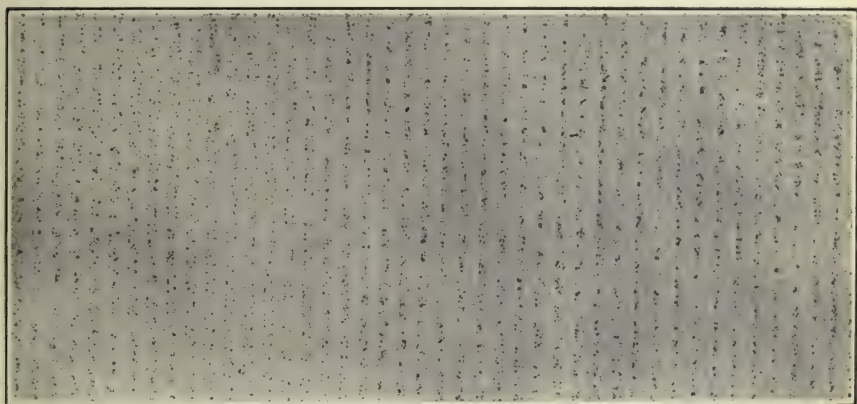


FIG. 2.—TEN TO THE INCH SOLID; STRAIGHT-CUT BURR. (HEMLOCK RUN No. 0.)

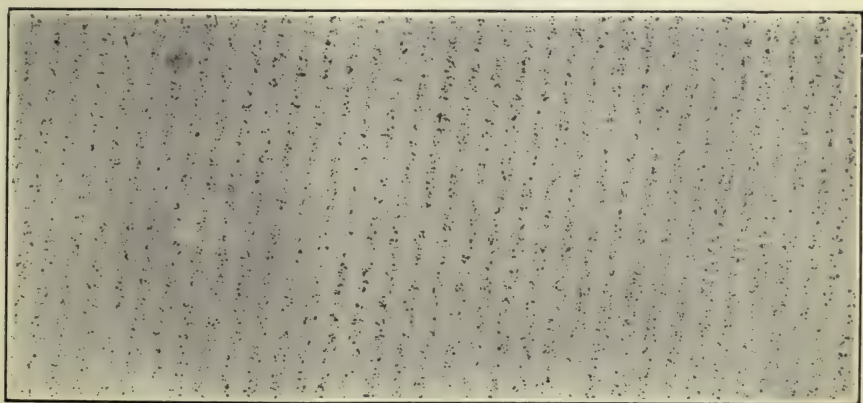


FIG. 3.—EIGHT TO THE INCH SOLID; SPIRAL BURR. (HEMLOCK RUN No. 43.)

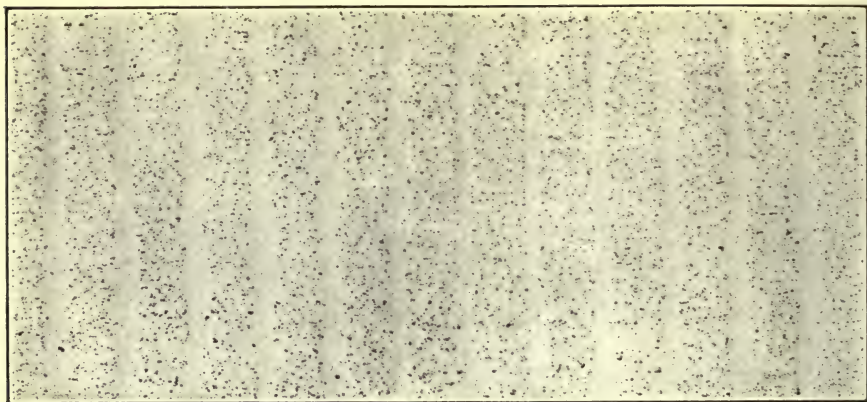


FIG. 1.—THREE TO THE INCH SOLID; STRAIGHT-CUT BURR. (HEMLOCK RUN No. 46.)

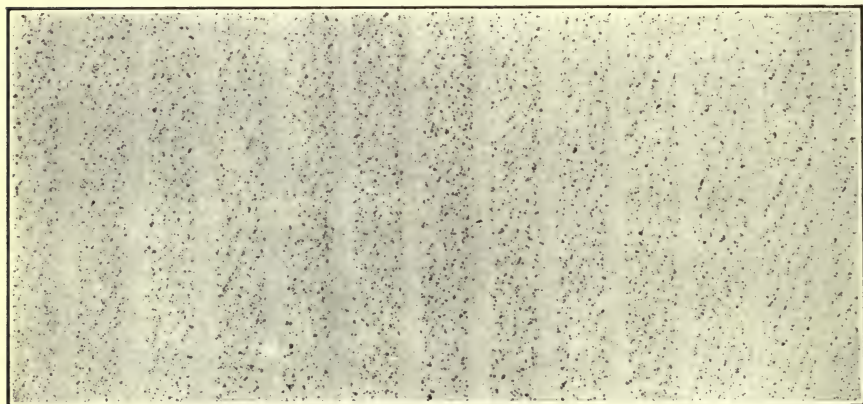


FIG. 2.—THREE TO THE INCH SOLID; STRAIGHT-CUT BURR. CUT OVER WITH TWELVE TO THE INCH SOLID; SPIRAL BURR. (HEMLOCK RUN No. 51.)

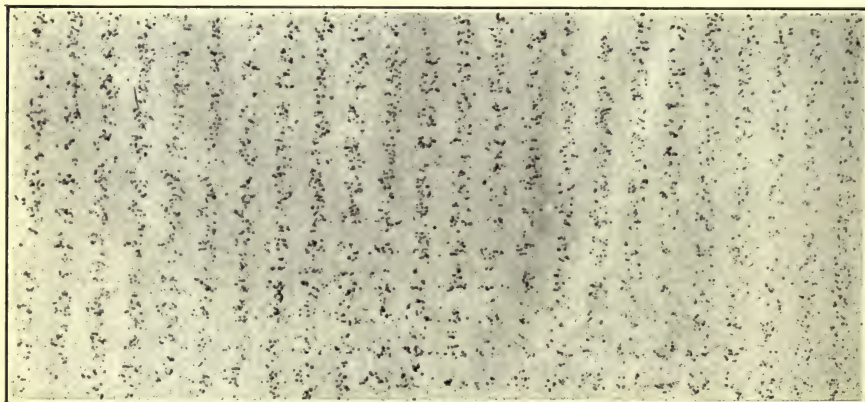


FIG. 3.—SIX TO THE INCH DIAMOND POINT BURR. (JACK PINE RUN No. 17-1.)

Swing cut-off saw.....	American Pulp and Paper Association.
3-ton scale.....	Paper (Inc.).
2 Ashton relief valves.....	Do.
1 pulp truck.....	W. A. Lounsberry & Co.
1 wood truck.....	Do.
1 54 by 27 inch pulp stone.....	Manufacturers' Paper Co.
12 42-inch screen plates.....	Union Screen Plate Co.
1 wet-machine felt.....	Albany Felt Co.
1 wet-machine felt.....	Appleton Woolen Mills.
1 set barker knives.....	Dowd Knife Works.
1 set sectional and solid burrs..	Ticonderoga Machine Works.

Plate I shows a portion of the pulp-making equipment. The grinder (Pl. I, fig. 2) has three pockets, the cylinders are 14 inches in diameter, and it is designed for a 54 by 27 inch pulp stone. Each of the grinder cylinders is equipped with a pressure gauge, and the pressure line between the triplex pumps and the grinder is provided with Ashton relief valves, which make it possible to obtain very uniform pressures up to 100 pounds per square inch.

A recording thermometer gives a record of the temperature in the grinder pit. From the grinder pit the pulp is passed through a mechanically agitated sliver screen, then pumped to a storage tank by means of a 6-inch centrifugal pump, and from there pumped to a centrifugal screen. A variable-speed motor direct connected to the screen makes it possible to obtain speeds of rotation from 400 to 600 revolutions per minute. Throughout the tests, however, the speed was maintained at 500 revolutions per minute. The plate in the centrifugal screen is perforated with holes 0.065 inch in diameter. The tailings from it are led by gravity to a 12-plate horizontal diaphragm screen, the plates of which are the Union Screen Plate Co's. type B, cut with 0.012-inch slots.

The good pulps from the centrifugal and the plate screens are united in the vat of the wet machine, which is direct connected to a variable-speed motor giving felt speeds ranging from 75 to 115 feet per minute. The wet machine is provided with a small triplex pump by which the cylinders connected to the press rolls are operated, the dryness of the pulp being determined by the pressure applied to the cylinders. A vacuum of from 10 to 15 inches, produced by a rotary suction pump, is maintained on the felt, and this, too, assists in obtaining the desired dryness of the pulp. The white water from the wet-machine vat is pumped back to the grinder sliver screen by a 4-inch centrifugal pump. White water from the felts is run directly to the sewer, as is also the white water from the felt suction. Plate I, figure 2, gives an idea of the general arrangement of the pulp-making machinery. A 40-inch swing cut-off saw and a Green Bay wood barker are used to prepare the wood.

METHODS EMPLOYED.

QUALITATIVE AND QUANTITATIVE TESTS.

In order to cover the field in a reasonable length of time, short tests ranging up to two hours in length were run. In these tests no attempt was made to cover every point, the object being to touch only such as were thought to have a marked effect on the quality of the product. The surface of the stone, the pressure on the grinder cylinder, and the peripheral speed of the stone were the variables which received most attention. No especial attention was given to economic considerations.

Tests were made with pressures of from 20 to 75 pounds per square inch on the cylinder, corresponding to from 8.2 to 30.8 pounds per square inch of pocket area. The speed of rotation of the stone was varied from 84 to 225 revolutions per minute, corresponding to a range in peripheral speed of from 1,173 to 3,150 feet per minute.

In studying the effect of the surface condition of the stone it was necessary to utilize burrs of many different types and designs. These ranged in fineness of cut from 12 to the inch to 3 to the inch. The style of cut differed also, spiral cut, diamond points, and straight cut being employed. The power applied to the grinder ranged from 87.3 to 520 horsepower, while the rate of production of bone-dry pulp varied from 1 ton to 7.3 tons in 24 hours. It should be understood that neither the two minimum nor the two maximum values were necessarily obtained from the same test. When the power applied to the grinder was 87.3 horsepower, for instance, it does not necessarily follow that the production was 1 ton per day. The horsepower consumption per ton under the given conditions was found to vary from 68.3 to 196 in 24 hours.

The samples of pulp obtained during the qualitative and quantitative tests were examined and commented upon by members of the advisory committee of the American Pulp and Paper Association, and those runs considered most promising were duplicated later in commercial tests.

COMMERCIAL TESTS.

TREATMENT OF THE WOOD BEFORE GRINDING.

All of the wood used in the tests was cut either in Wisconsin or Michigan and was representative of the species. In some cases the wood was secured directly from the forest, while in others it was shipped to the laboratory from near-by mills. Upon arrival at the laboratory the logs were closely piled on skids. An attempt was made to keep the material green by painting the ends with paraffin, but this proved unsatisfactory, because the paraffin peeled off. The wood tested was taken directly from the piles for all tests up to and includ-

ing run No. 52 of the hemlock series, with the exception of runs Nos. 49, 50, and 51, the wood for which was soaked in the pond for approximately two months before being used. The only jack pine soaked was that used in the commercial test on seasoned wood of that species—run 14. The jack pine and spruce used in tests on mixed pulps were in all cases dry before grinding. The wood for the tests was prepared approximately 2 cords at a time, sawed into 2-foot lengths, barked, weighed, and piled up for the grinding process.

To determine accurately the yield, the bone-dry weight per cubic foot of wood, as well as the percentage of moisture present, was determined in each commercial test. All weighings were made in 500 or 1,000 pound lots, and the wood was used as soon as ground.

No attempt was made to remove knots or punky portions of the wood. In fact, all of the tests were carried on in accordance with the usual commercial practice.

GRINDING.

Before commencing the grinding tests an impression of the surface of the stone which had been selected was taken by means of a piece of carbon paper and a sheet of coated paper. This impression was later photographed, as shown in Plates II and III. In these the black dots represent projecting points and the white portions between them depressions in the stone. The surface shown in Plate III, figure 2, is particularly interesting, since it is the result of dressing with two different kinds of burrs.

Before starting the tests the recording thermometer and all of the other recording instruments were placed in operation. The pockets of the grinder were filled, the pressure adjusted to the proper value, and the grinder started.

For the purpose of check and control, regular readings were taken of the various switchboard instruments, the indicating tachometer, the pressure gauges, and the recording thermometer. On short tests up to 2 hours in length these readings were recorded at 5-minute intervals, but on longer tests the interval was increased to 15 minutes. The speed, pressure, and other variables were maintained as nearly constant as possible. For instance, when one of the grinder pistons was raised the speed was brought back to the desired value by manipulation of the rheostat controlling the motor armature voltage.

During the qualitative and quantitative tests the pulp stone did not have an opportunity to heat up, and, in consequence, some of the data on power consumption and production may be more or less questionable. In the commercial tests, however, all of which were made under the hot-grinding process, the stone was brought up to a high temperature, which was maintained throughout the run; consequently these more nearly approximate commercial conditions.

LOSSES IN GRINDING.

To determine approximately the losses occurring in the conversion of wood to pulp, the bone-dry weight of screenings obtained from a known amount of bone-dry wood was determined. The loss in the white water was then taken as the difference between the bone-dry weight of the wood ground and the bone-dry weight of the pulp secured plus the screenings.

FIBER STUDY.

During each test the character of fiber obtained was examined by means of an apparatus for microscopic study. This consists of an ordinary stereopticon provided with a specially constructed carrier for microscopic slides. Samples of wet pulp were taken from the wet-machine vat and slides were made by first removing the water by drying, then staining with Bismarck brown, and moistening with glycerine. The mixture of glycerine and fiber was teased out to cover the area of an ordinary microscopic cover glass, which was placed over the mixture. Evaporation or leakage was prevented by means of a thin strip of shellac around the edge of the cover glass.

With this apparatus it was also possible to compare different samples of pulp with the commercial standards used, the latter being selected from a large number of samples submitted by American manufacturers of ground-wood pulp.

CALCULATION OF RESULTS.

To give a clear understanding of the method employed in calculating the various items in connection with a test, all the calculations for a representative run, No. 50, Table 4, are given here. Considerable data taken during the tests have been eliminated from the compilation, since they have no direct bearing on the study.

The test mentioned required 3.42 hours to complete, and during that time 3,388 pounds of hemlock wood were ground. For comparison all of the figures on weight of wood were brought to a bone-dry basis. The bone-dry weight of wood was secured by drying a known volume of wood to constant weight and calculating the weight per cubic foot. By calculating the bone-dry weight of a log of measured volume and subtracting this amount from the actual weight of the log, the moisture content of the wood was determined. The bone-dry weight of this wood, per cubic foot, was 24.84 pounds, and the moisture content was 46.5 per cent; consequently the amount of bone-dry wood ground was 53.5 per cent of 3,388, or 1,810 pounds. This is equivalent to 72.9 cubic feet of solid wood ground during the

given period; or, in other words, the grinding was carried on at the rate of 512 cubic feet of solid wood in 24 hours.

The amount of wet pulp obtained during the test was 3,795 pounds, and this upon analysis was found to have a moisture content of 60.22 per cent. Consequently, 1,507 pounds of bone-dry pulp were obtained during the period of test, corresponding to a production of $\frac{24}{3.42} \times \frac{1507}{2000}$ or 5.3 tons in 24 hours. To grind the wood and produce this pulp it was necessary to apply to the grinder motor power which averaged 338 kilowatts. This value was obtained by dividing the total number of kilowatt hours used, as given by a watt-hour meter, by the length of the test in hours. Figure 1 shows a section of a wattmeter record obtained during this test, and illustrates how the power used by the grinder varied upon the removal of the load from one of the grinder pockets. The entire recording wattmeter curve was averaged by means of a planimeter, in order to check the value of power consumed as given by the watt-hour meter.

By using curves which show the losses in the motor it was found that 15.3 kilowatts were required to supply the heat losses in the grinder-motor armature and 7.1 kilowatts to supply the stray power losses, making a total of 22.4 kilowatts lost in converting the power from electrical to mechanical. This amount subtracted from 338 kilowatts gives 315.6 kilowatts which were furnished to the grinder pulpstone, and 315.6 divided by 0.746¹ gives the value of 422½ horsepower applied to the grinder. In order, then, to obtain 5.3 tons of bone-dry pulp in 24 hours it was necessary to apply to the grinder over that period 422½ horsepower, or the horsepower consumption per ton was 422½ divided by 5.3, or 79.7 horsepower per ton in 24 hours.

The yield from 100 cubic feet of solid wood was obtained by dividing the amount of pulp produced during 24 hours, 5.3 tons or 10,600 pounds, by the number of hundreds of cubic feet of wood ground in 24 hours, viz, 5.12. The result is 2,070 pounds.

The average temperature of grinding was determined by reading the recording thermometer every five minutes, adding these values, and dividing by the total number of readings.

COMPARISON OF YIELDS.

Much importance is attached to the amount of pulp obtained from a cord of wood, because this represents the efficiency of conversion. Commercial practice in the manufacture of spruce ground wood requires a yield of approximately 2,300 pounds per cord of

¹ A horsepower is equivalent to 0.746 kilowatt.

rossed wood, or 1,800 pounds per cord of rough wood. The average yields which have been obtained for hemlock and jack pine, together

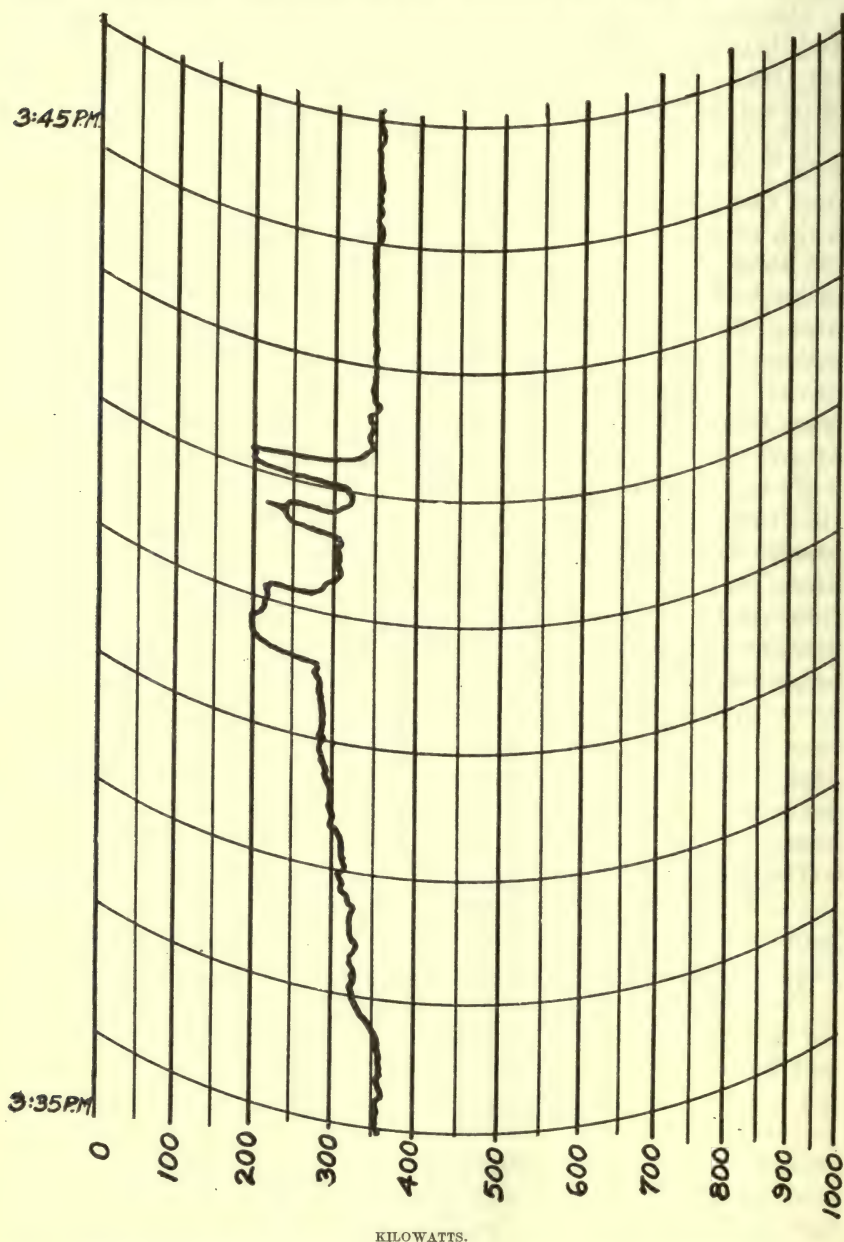


FIG. 1.—Portion of chart from recording wattmeter, showing decreased power consumption after removal of load from pocket and pocket binding.

with those obtained for two different shipments of spruce, are given in Table 1.

TABLE 1.—*Average yields from spruce, hemlock, and jack pine.*

Species.	Weight per 100 cu. ft. bone-dry.	Yield per 100 cu. ft. of solid wood.	Efficiency of conversion.
	<i>Pounds.</i>	<i>Pounds.</i>	<i>Per cent.</i>
Spruce.....	2,840	2,480	87.3
Do.....	2,270	2,000	88.2
Hemlock.....	2,480	2,100	84.8
Jack pine.....	2,540	2,200	86.7

One hundred cubic feet of solid wood was selected as the basis of yield, since it eliminates the variable loss in barking, and represents fairly well the amount of solid wood in a rossed cord. The yield, as will be seen, is directly proportional to the bone-dry weight of the wood. The loss in conversion has been found to range between 12 and 15 per cent of the original weight of the bone-dry wood. Approximately 6 per cent can be accounted for in the white water and 1 per cent in screenings. The manner in which the remaining losses occur has not been determined, but will be studied in future tests.

The storage capacity for white water in the laboratory was very limited, and this may to some extent account for the low yields. The continuous use of the white water and the use of save-alls would undoubtedly tend to increase the yields and result in saving a great deal of fine pulp.

The loss in barking jack pine and hemlock, so far as has been determined up to the present time, is practically the same as the loss in the barking of spruce. There are a great many knots in both hemlock and jack pine, and it is possible that this may cause a somewhat greater loss in barking these species. However, on account of the small amounts of the various woods used, no reliable data on loss were obtained.

FACTORS WHICH INFLUENCE QUALITY AND PRODUCTION.

SPEED AND PRESSURE.

The effect of speed on the quality of pulp can best be illustrated by the magnified fibers shown in Plates VII and VIII. In grinding these pulps the pressure and surface of the stone were maintained constant, and the speeds were, respectively, 100, 150, and 200 revolutions per minute for the hemlock, and 152 and 205 revolutions for the jack pine. There is little difference in the fibers ground under these different conditions of speed; especially those run at 150 and 200 revolutions per minute. Speed probably has very little effect on the quality of pulp. With satisfactory pressure and surface of stone, it is possible to obtain good grades of pulp at any speeds within reasonable limits. Commercially, it is practically impossible to main-

tain the speed constant at all times. When the pressure on a pocket is removed the speed is bound to rise considerably, especially when the water wheels or turbines are operated without a governor.

When hemlock wood was ground at low speed and low pressure it was impossible to obtain anything more than a powder. Also when this wood was ground at low pressure and high speed the product was extremely short, but the pressures at which these results were obtained are considerably lower than those ordinarily employed commercially, and the results have little significance. If the stone is what is ordinarily called sharp, it is necessary to use a lower pressure, and when dull, a higher one, but it is impossible to obtain the same quality of pulp under both conditions. Speed and pressure affect quantity rather than quality, and by the proper adjustment of both the maximum efficiency of grinding is attained. If a certain speed is selected there must be a corresponding pressure which will yield the greatest amount of pulp in 24 hours with the least consumption of power.

By the term "constant pressure," wherever used in this report, is meant constant pressure on the grinder cylinders. The pressure per square inch of wood in contact with the grinding surface varies considerably, chiefly with the size of wood ground and the area of the pocket. Again, the length of the wood is not at all a constant quantity, and this, too, can only result in a variable pressure per square inch of wood. The pressure of the wood on the stone varies throughout certain limits with any pressure on the grinder cylinder, and the ranges of pressure of the wood on the stone are raised or lowered by raising or lowering the cylinder pressure. This pressure variation, however, can hardly be controlled commercially, and therefore has not been considered in the tests discussed in this report. There is also more or less pressure variation due to binding of wood in the pockets, and this, too, is difficult, if not impossible, to control. Figure 1 shows a measure of the power applied to the grinder. The effect of pocket binding and the withdrawal of pressure from one of the pockets will be noted. At one end of this chart the power consumed is approximately 360 kilowatts, falling off gradually to 280 kilowatts, due to pocket binding. After raising the pistons and readjusting the wood in the different pockets, the power to the grinder motor had to be increased to 350 kilowatts on account of the added load produced by eliminating the pocket binding.

SURFACE OF STONE.

The most efficient grinding condition is one where there is a maximum amount of grinding surface, and still a sufficient amount of depression in the stone to allow for the carrying away of the ground wood, or, as this is commonly called, for the clearing of the stone.

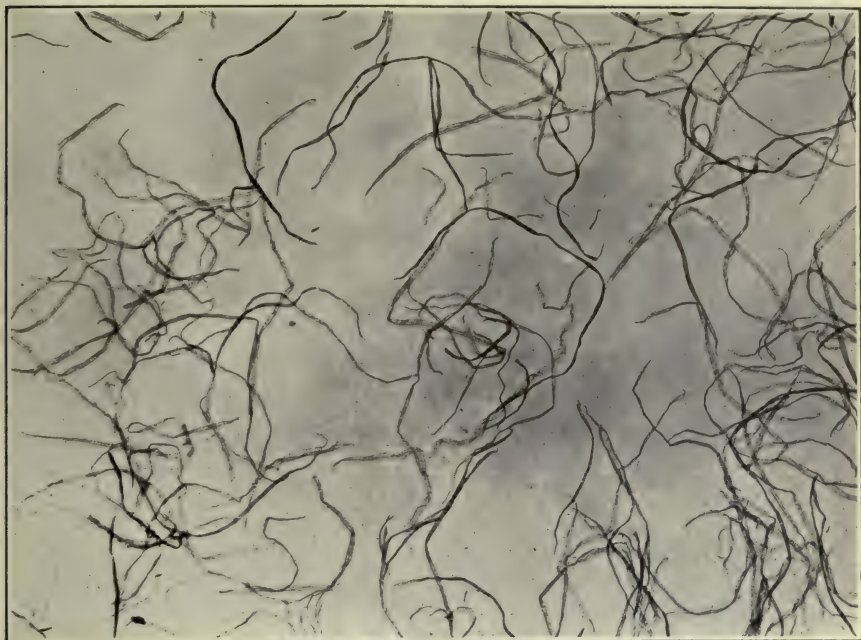


FIG. 1.—SPRUCE SULPHITE STANDARD.

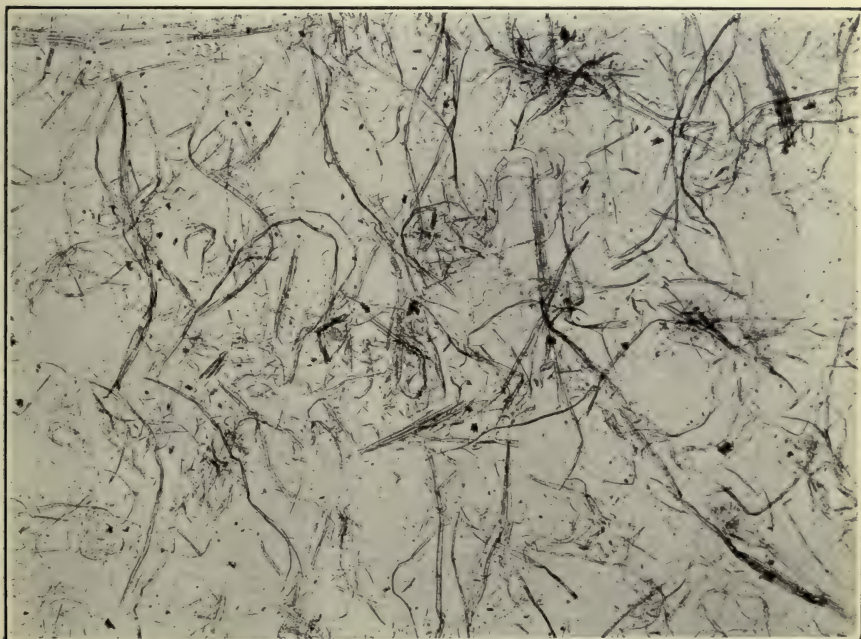


FIG. 2.—SPRUCE GROUND WOOD, NO. 1 STANDARD.



FIG. 1.—SPRUCE GROUND WOOD, NO. 2 STANDARD.



FIG. 2.—SPRUCE GROUND WOOD, NO. 3 STANDARD, COARSE GROUND.



FIG. 1.—SPRUCE GROUND WOOD, NO. 4 STANDARD, MEDIUM GROUND.



FIG. 2.—SPRUCE GROUND WOOD, NO. 5 STANDARD, FINE GROUND.

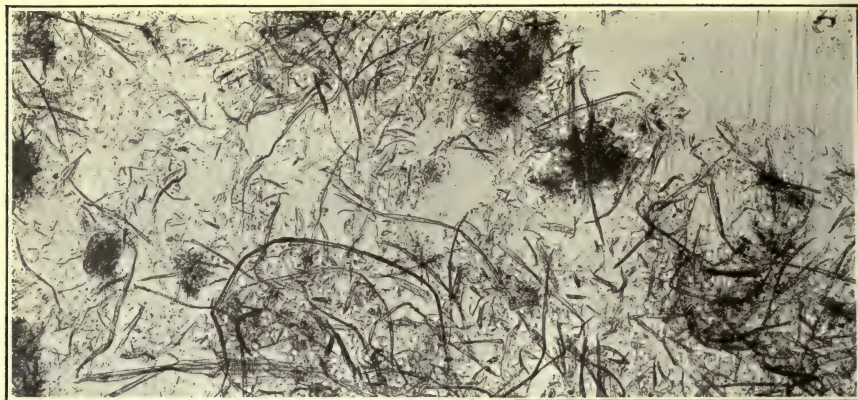


FIG. 1.—100 R. P. M. (Run No. 35.)

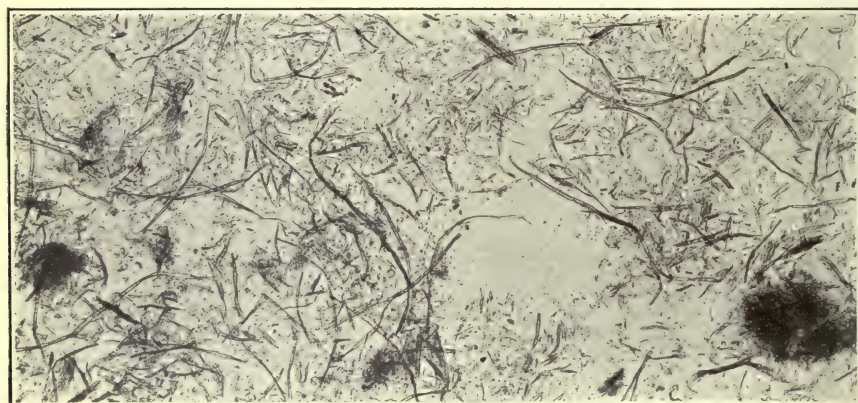


FIG. 2.—150 R. P. M. (Run No. 36.)

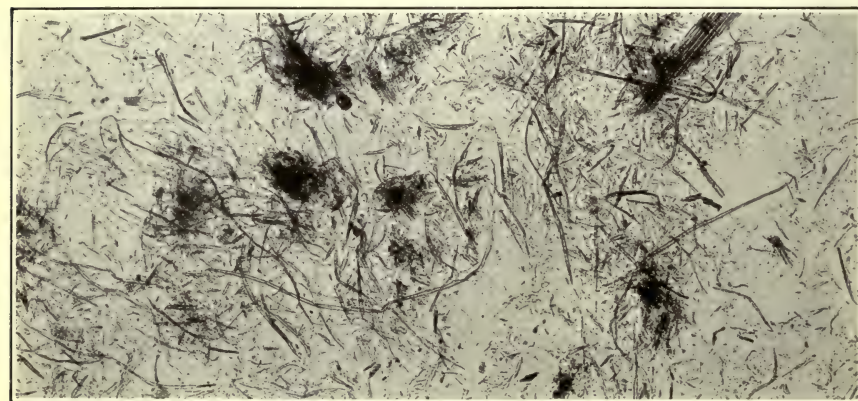


FIG. 3.—200 R. P. M. (Run No. 37.)

COMPARISON OF HEMLOCK PULPS GROUND AT DIFFERENT SPEEDS.



FIG. 1.—152 R. P. M. (Run No. 19.)



FIG. 2.—205 R. P. M. (Run No. 20-1.)

COMPARISON OF JACK PINE PULPS GROUND AT DIFFERENT SPEEDS.

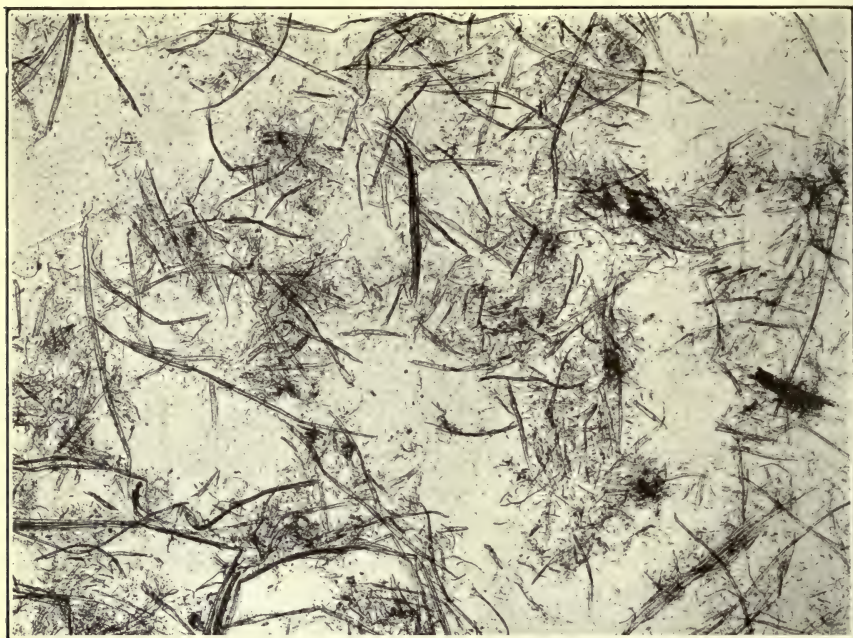


FIG. 1.—JACK PINE GROUND WOOD. (RUN NO. 14-1.)



FIG. 2.—JACK PINE GROUND WOOD. (RUN NO. 13-1.)



FIG. 1.—JACK PINE GROUND WOOD. (RUN NO. 7-1.)

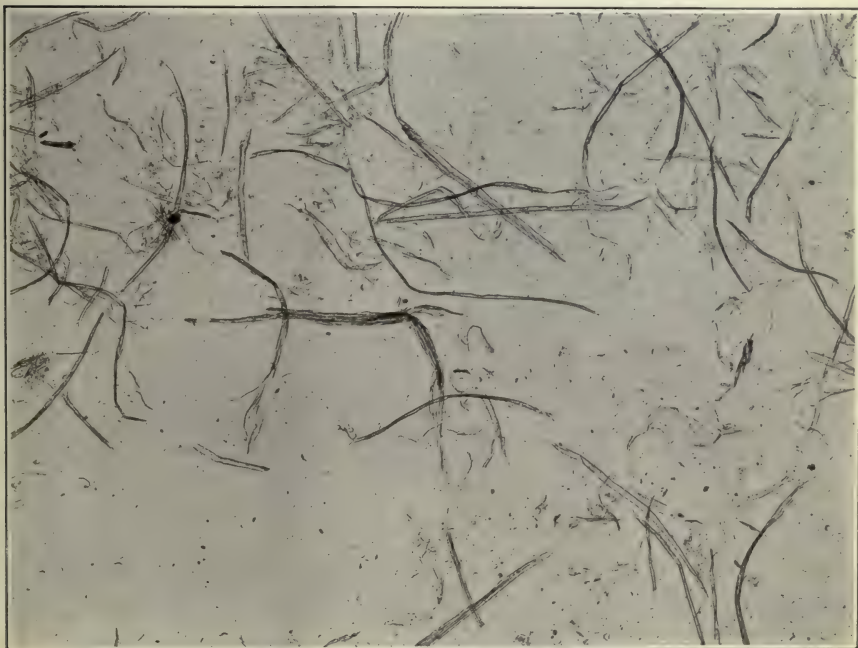


FIG. 2.—JACK PINE GROUND WOOD. (COMMERCIAL RUN NO. 24.)

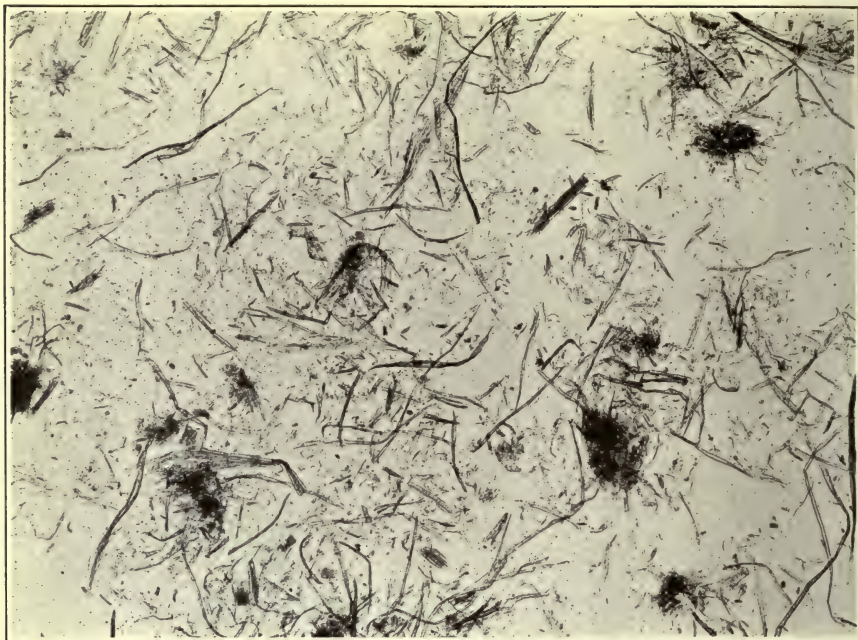


FIG. 1.—HEMLOCK GROUND WOOD. (RUN No. 41.)

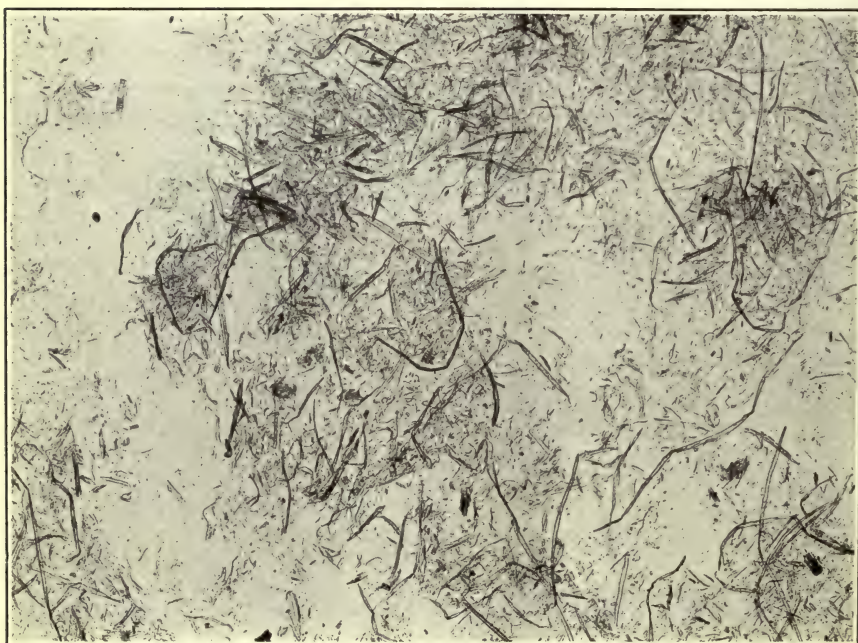


FIG. 2.—HEMLOCK GROUND WOOD. (COMMERCIAL RUN No. 14-1.)



FIG. 1.—HEMLOCK GROUND WOOD. (COMMERCIAL RUN NO. 8.)

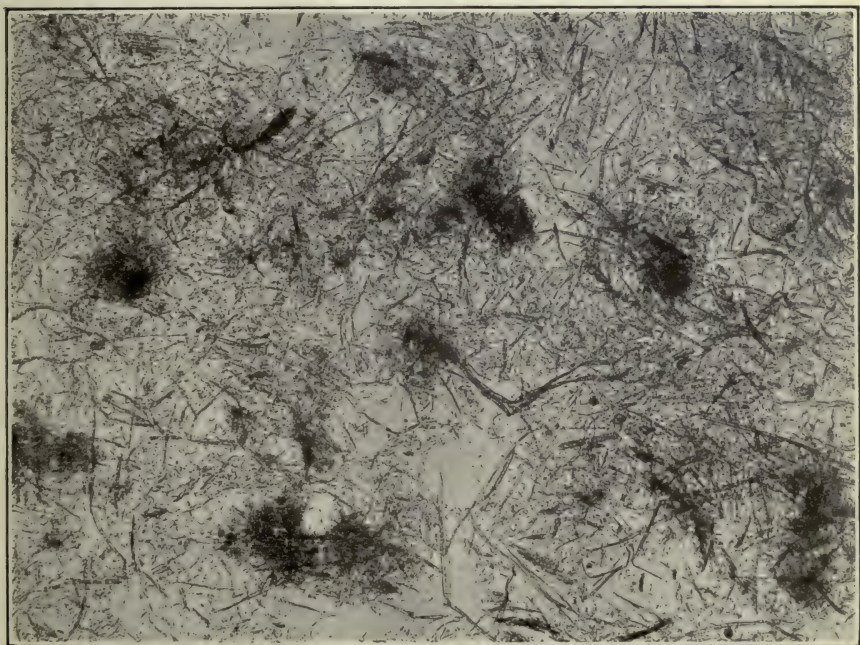


FIG. 2.—HEMLOCK GROUND WOOD. (RUN NO. 2.)



FIG. 1.—HEMLOCK GROUND WOOD. (COMMERCIAL RUN NO. 30)

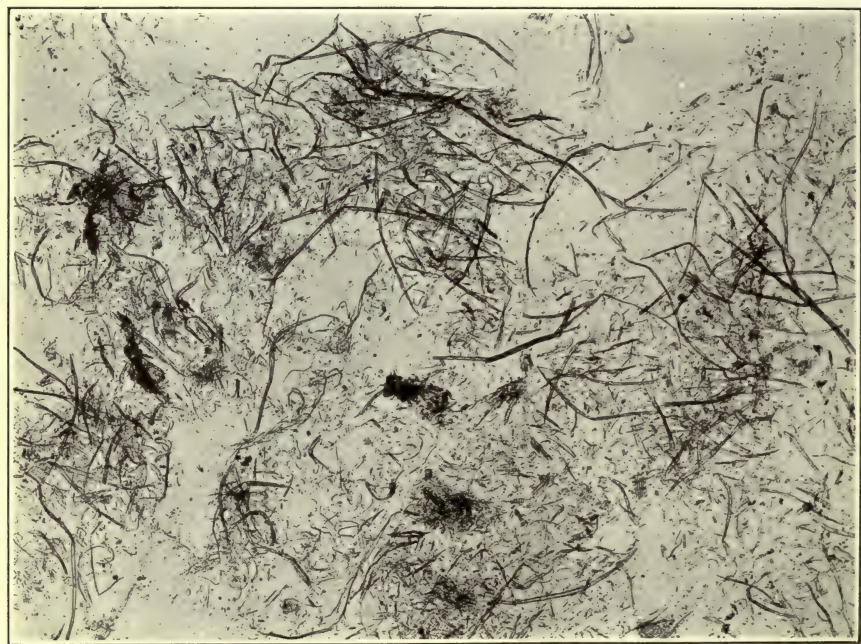


FIG. 2.—HEMLOCK GROUND WOOD. (COMMERCIAL RUN NO. 50.) USED IN ACCOMPANYING PAPER SAMPLE.

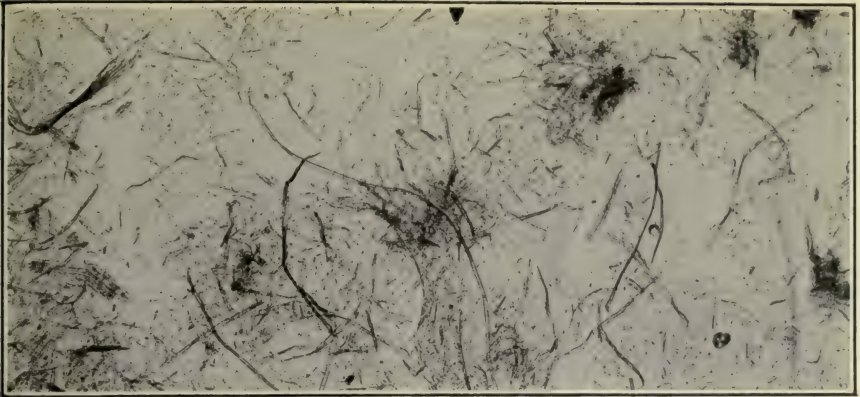


FIG. 1.—GROUND-WOOD PULP, ONE-THIRD SPRUCE, TWO-THIRDS HEMLOCK. (RUN No. 46A.)

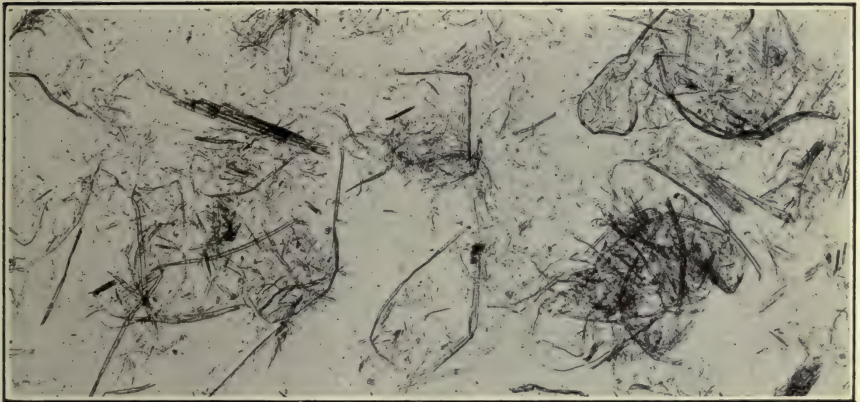


FIG. 2.—GROUND-WOOD PULP, ALL HEMLOCK. (RUN No. 46B.)

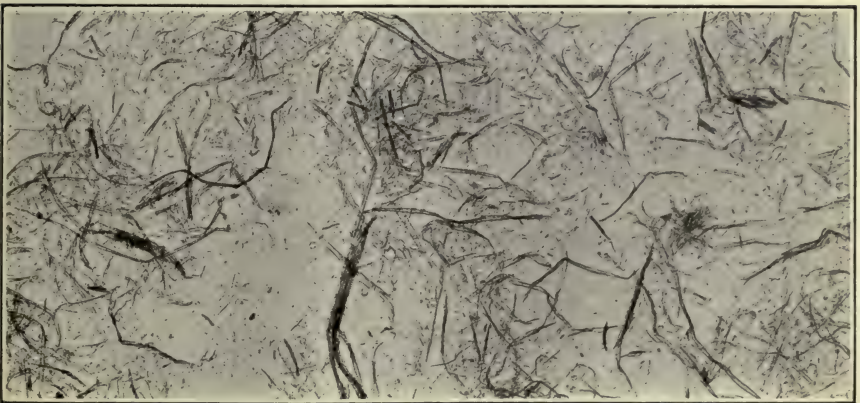


FIG. 3.—GROUND-WOOD PULP, ALL SPRUCE. (RUN No. 46C.)

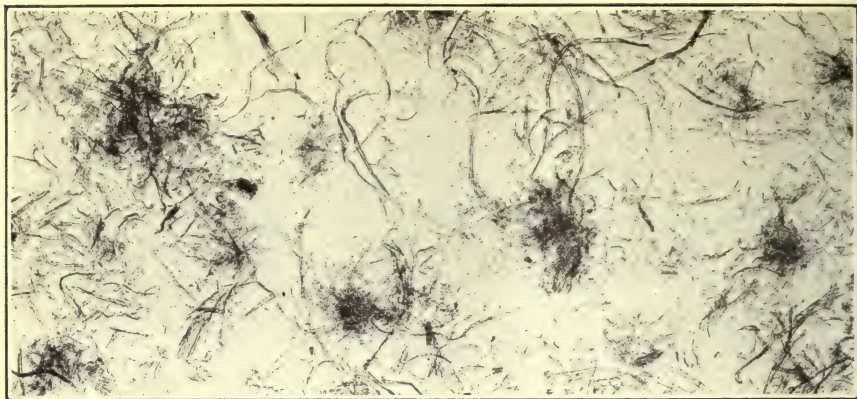


FIG. 1.—ONE-THIRD SPRUCE, TWO-THIRDS HEMLOCK. (COMMERCIAL RUN NO. 46A.)

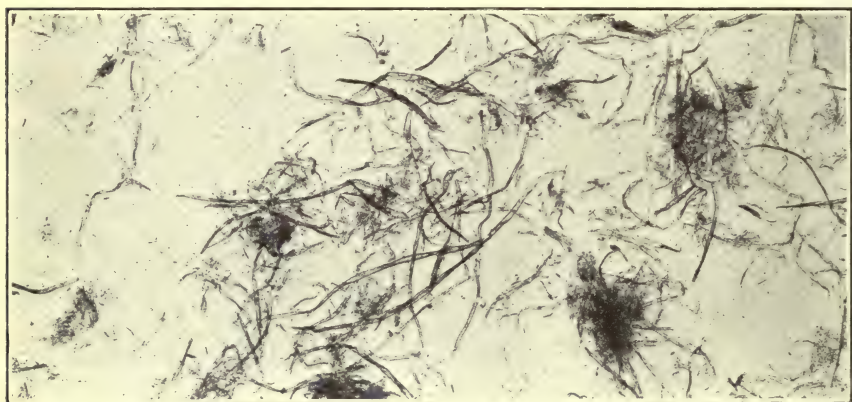


FIG. 2.—ONE-THIRD HEMLOCK, ONE-THIRD JACK PINE, ONE-THIRD SPRUCE. (COMMERCIAL RUN NO. 51.)

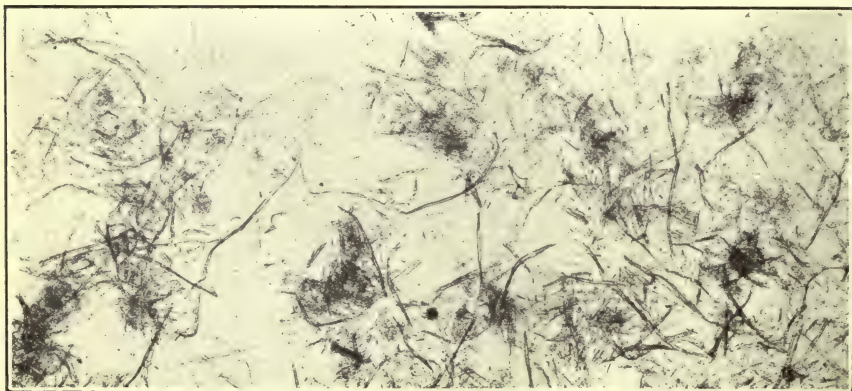


FIG. 3.—ONE-THIRD JACK PINE, TWO-THIRDS HEMLOCK. (COMMERCIAL RUN NO. 52.)

MIXED GROUND-WOOD PULPS USED IN ACCOMPANYING PAPER
SAMPLES.

Throughout the experiments, particularly the commercial tests, it was found that the pulp of same appearance as regards fiber and of the same apparent strength can be obtained by using burrs of widely different design and fineness of cut, provided the grit of the stone is in each case the same. For example, during the commercial tests the stone was burred at different times with different types of burrs, and the grinding in each case was found to require the consumption of the same amount of power. The production per day was the same also, provided the grit was brought to the same condition of sharpness and the other variables were kept constant.

During some of the preliminary tests the surface of stone was dulled with a fire brick, as is often done in mills. This appears to have been unnecessary; in fact, the result is detrimental rather than beneficial.

Better pulp was obtained and the production was increased slightly by crushing the tops of the ridges formed in burring by means of a solid, smooth bush roll. This method does not smooth off the individual particles of sand on the stone as dulling with the brick does, but rather sharpens them. During the tests conducted on mixed woods a surface obtained by the use of a three-to-the-inch straight-cut solid burr and a 12-cut spiral burr was used. The stone was first dressed with a three-to-the-inch burr, forming grooves in the stone approximately one thirty-second inch deep; then the portion of the stone between these depressions was roughed with a 12-cut spiral burr. This caused the grit to stand out and gave a maximum of useful grinding surface. The pulp obtained with this surface was almost entirely free from shives, and the fibers were long and fine. The surface of stone used during these tests is shown in Plate III, figure 2.

A great deal of experimentation still remains to be done, not only with burrs of different cut and design, but more especially with stones of different grits, since it appears that the grit is more responsible for the quality of pulp obtained than any other variable feature in its production.

Where the pulp stone is deep burred, however, the grit is not so important a factor of quality. When the power consumed in making a ton of bone-dry pulp is as low as 50 to 60 horsepower, the added production which must be secured to bring the power to this low value is obtained through the action of the ridges on the stone and not through the grit. When it is desired to manufacture a pulp of high quality, however, it is the grit of the stone and the manner of raising it which must be considered. The type of burr used and the depth of dressing both influence production, but it is only the latter that influences the quality. When the pulp stone has been dressed so as to provide just sufficient depression to carry away the ground

wood a high-grade pulp will be produced, providing the grit of the stone is suitable, irrespective of the style of the burr and within reasonable limits of the pressure used. It is not impossible to conceive of an artificial stone which could be used continually without burring, which would clear itself without having depressions or ridges, and which would have the correct size and kind of grit to give the maximum production and best quality.

TEMPERATURE.

The temperature of grinding, it is said, has much to do with the quality and quantity of pulp obtained, and many manufacturers insist that it is impossible to secure a tough, strong fiber with anything but the hot grinding process. It was noted in the experiments that the rate of production was not nearly as great at a low temperature as it was after a high one was reached. However, it was impossible to detect microscopically any difference in the fibers themselves. As has been said, the only observations made on cold grinding were while the stone was warming up, and on this account it is impossible to say definitely what particular advantages or disadvantages, if any, lie in the hot grinding process.

UNDETERMINED FACTORS.

Since the experimental work on hemlock, jack pine, and spruce was started a number of factors which more or less influence the quality and the rate of production of pulp have made themselves evident. These are the rate of growth of the wood, moisture content of the wood, size of wood ground, temperature of grinding, the thickness of stock in the grinder pit, and the grit of the pulp stone, the last undoubtedly being the most important. All of these variables will be studied in future experiments, though the grit of the pulp stone is the one which will probably receive the greatest attention. It is doubtful whether this very important item in the production of ground wood has been given sufficient consideration by manufacturers.

MICROSCOPIC COMPARISON OF EXPERIMENTAL PULPS AND COMMERCIAL STANDARDS.

STANDARD PULPS.

Since it was necessary to have some means of comparing the experimental pulps produced with commercial products, portions of the samples obtained from manufacturers were photographed, in an endeavor to classify the fibers according to their quality. It was found that there is a more or less regular grading of the material from long, fine fiber to pulp which is almost a powder. It is probable

that each of the various grades of pulp has its particular use in certain qualities of paper, though no attempt has been made to classify them according to uses. The standards selected are shown in Plates IV, V, and VI; in each case the fibers are enlarged 15 times. For the purpose of comparison the spruce sulphite standard fiber is shown in Plate IV, figure 1, and the No. 1 spruce ground-wood standard in figure 2. It is indeed very seldom that a sample of pulp is obtained which corresponds to the No. 1 spruce ground-wood standard. As will be noted, the fibers are very similar to the sulphite fibers, although there is a certain amount of short material and coarse fiber present which does not occur in the sulphite pulp.

Plate V shows the No. 2 ground-wood standard and No. 3 coarse ground-wood standard. The No. 2 differs from the No. 1 standard only in the amount of short fiber and coarse fiber present, the No. 2 having larger amounts of these two kinds. The coarse standard needs no description. It is a kind of fiber often made by mills during their low-water periods in order to maintain production with less power consumption.

The No. 4, medium ground-wood standard, and the No. 5, fine ground-wood standard, seem to follow in logical sequence those previously given. The No. 4 standard has the appearance of being a mixture of No. 3 and No. 5. The No. 5, as will be noted, contains an extremely small amount of fiber and is composed largely of dust and short-fiber particles.

Table 2 gives the data furnished by the manufacturers of the various pulp samples selected as standards. These data, for the most part, are only approximate, but they will serve to give some idea of the conditions under which the material was produced.

TABLE 2.—*Conditions of manufacture of spruce ground-wood standards.*

Number of stand-ard.	Make of grinder.	Number of pock-ets.	Size of cylinders.	Pressure on cylin-ders.	Equivalent pres-sure on 14-inch cylinder.	Kind of stone.	Kind of burr.	Size of stone.	
								Diameter.	Face.
1	Friction Pulley & Machine Works.	3	16	35	45.7	Lombard.....	Washers.....	54	27
2	Carthage Machine Co.....	3	16	40	52.2	Lombard and Manufacturers' Lombard.....	Solid spiral cut 8 to 1 inch.	54	26
3	Dayton Globe Iron Works....	3	18	45	74.4	Lombard.....	Diamond point cut 6 to 1 inch.	54	27
4	Carthage Machine Co.....	3	14	60	60.0	Empire.....	Straight cut 7 to 1-inch.	54	25½
5	Friction Pulley & Machine Works.	3	10	90	45.9	Greeley, New Castle.	Diamond point 7 to 1-inch and 5 to 1-inch.	54	18½

TABLE 2.—*Conditions of manufacture of spruce ground-wood standards—Continued.*

Number of stand-ard.	Make of grinder.	Revolutions per minute of stone.	Peripheral speed of stone.	Horsepower per ton.	Temperature of grinding.	Size of screen slots.	Grade of paper.
1	Friction Pulley & Machine Works.....	180	2,545	90	° F. 160	.012	Do not make paper.
2	Carthage Machine Co.....	190 to 200	2,686 to 2,827	75	175	.012	Poster.
3	Dayton Globe Iron Works.....	145	2,050	1 300		.010	News.
4	Carthage Machine Co.....	270	3,817	100		.075	Do.
5	Friction Pulley & Machine Works.....	220	3,110	70	Cold.	.011	Specialties. Some coated, requiring a soft and fine pulp.

¹ To grinder.

JACK PINE PULP.

Plates VIII, IX, and X show six samples of ground-wood fiber obtained from jack pine. The data taken during these tests are given in Table 4. There is a very striking likeness between the fiber obtained from jack pine and that from spruce. Especially is this true of the fiber secured by using the natural grit of the pulp stone without any burring. Tests Nos. 7-1, 19, and 13-1 were run by using an excessive amount of power and by sacrificing the rate of production. This wood was ground, however, in the dry state, and unquestionably the same quality of fiber could be obtained with increased production and lower horsepower consumption per ton if it was soaked or steamed. The fibers obtained in tests Nos. 20-1, commercial run No. 14-1, and commercial run No. 24 compare favorably with the No. 4 standard, and the production and power consumption are more nearly those obtained commercially. There is more short material than is found in the No. 1 or No. 2 standards, but still the fiber is long and fine, and appears to have considerable strength. The illustrations show some of the better fibers obtained. Of course pulp has been made in the laboratory which was fully as fine as that shown as the No. 5 standard. Some has been made also which is fully as coarse as the No. 3. However, this is rather the exception than the rule.

The jack pine fiber shown in Plate X (run No. 7-1) was the most pleasing in appearance when in the pulp lap, and was generally considered to possess the best quality. The rate of production, however, was so very low, and the horsepower consumption so high, that it has no commercial importance.

HEMLOCK PULP.

Plates XI, XII, and XIII show hemlock fibers which were obtained during the tests. Undoubtedly the most notable feature is the large amount of fine material present as compared with the jack pine samples. Hemlock grinds short and fine to a far greater extent than either spruce or jack pine. While there are a great many long fibers present, they are not sufficient in number to give a strong and tough pulp. A certain amount of short material is necessary, however, for news paper, and it is this material which gives a good finish. The finish on the paper made from the hemlock pulp, commercial run No. 8, was exceedingly good; in fact, the superintendent of the mill where the sample was run pronounced it better than the standard news. It will be seen that there is a regular gradation in the length of fibers from the long to the almost powder form. On certain of the illustrations of hemlock fiber black spots composed of a great deal of fine fiber will be noticed. These are the result of drying the pulp before making slides, it being impossible entirely to beat out the fiber after drying. When the material was in the form of pulp laps there was no marked difference in the pulps. However, as with the jack-pine samples, only the better grades of pulp obtained are shown. The sample of pulp illustrated in Plate XIII, figure 2 (commercial run No. 50), is fairly representative of the pulp which it is possible to make from hemlock under the conditions described.

MIXED PULPS.

Plate XIV shows three photomicrographs of fibers obtained under exactly the same conditions of pressure, speed, and surface of stone. The temperature and other minor variables were also kept as nearly alike as possible.

Figure 1 shows fibers obtained by grinding hemlock in two of the grinder pockets and spruce in the third (run 46a). Upon determination it was found that the pulp contained 34 per cent spruce and 66 per cent hemlock. Figure 2 shows hemlock fiber obtained under the same conditions (run 46b) as the first test, and figure 3 shows a number of spruce fibers (run No. 46c). The hemlock fiber is considerably shorter than the spruce and there are more shives present. In the composite sample the hemlock is decidedly in evidence.

Plate XV shows three fibers obtained by grinding different woods in different pockets of the pulp grinder. Commercial run No. 46a is composed of a mixture one-third spruce and two-thirds hemlock; commercial run No. 51 is composed of one-third jack pine, one-third spruce, and one-third hemlock; commercial run No. 52 of one-third jack pine and two-thirds hemlock. All of these pulps when in the

lap appeared to be a very good quality; in fact, it will be seen that the fiber of which they are composed is of good length and that there is not nearly as much short material present as there is in the samples of hemlock pulp.

Commercially it would be possible to obtain better mixed pulps by grinding the different woods in separate grinders and preparing the stones so as to obtain the best quality of pulp from each wood.

It has been found, by comparing the samples submitted by American manufacturers with the standards chosen, that 5 per cent can be classed as No. 1 pulp, 12 per cent as No. 2 pulp, 12 per cent as No. 3 pulp, 61 per cent as No. 4, and 10 per cent as No. 5. Comparison of the experimental pulps with the commercial standards shows that mixed pulps particularly compare well with the No. 4 standard, for which there is evidently the greatest demand.

SAMPLES OF PAPER PRODUCED.

In order to determine the adaptability of the pulps obtained in the experiments to the manufacture of paper, a number of test paper runs were made with the pulps which gave greatest promise. Samples of the paper obtained accompany this report. All of the sheets of news paper were made on a machine in the Port Edwards mill of the Nekoosa-Edwards Paper Co. This machine is 116 inches wide, and the sheet produced, trimmed, was 109 inches. The machine was operated at a speed of 465 feet per minute, and no changes were made in weight of sheet or speed after the beginning of the test; in fact, throughout the runs the conditions were maintained as nearly constant as possible. The finish on the paper was obtained by passing the sheet nine times through a 12-roll calender stack. In each case three 1,500-pound beaters of stock were run into sheet in order to have the test continuous over sufficient time to give an idea of its operation on the paper machine. It was intended to by-pass the Jordan engine, but this being impossible, the stock was passed through the engine and the roll set up only slightly.

Running changes were made in each test, and no difference was found with any of the sheets excepting jack pine. This material was somewhat pitchy, and after an hour's run it was necessary to remove the dandy, since it began to pick up stock. All of the papers were free on the wire and caused no trouble whatever.

The samples of paper containing spruce were made up for the purpose of comparison. It will be seen that, with the exception of color, the sheets differ little, and it is reasonable to suppose that the color could be improved. Allowance should be made for the appearance of the sheets as regards brown shives, these being due to the hemlock sulphite used, and not to the ground wood. Data on the beater "furnish" for the various papers are given in Table 10. Table 3 gives a comparison of strength of the various sheets.

The samples of butcher's manila and No. 2 white manila given were made by the Nekoosa-Edwards Paper Co., the furnish being shown in Table 10. These samples are meant merely to give an idea of what can be obtained when mixtures of hemlock and spruce are used.

The strength of all of the sheets, with the exception of the one made up of hemlock sulphite and jack-pine ground wood, compares well with standard news paper. The paper from run No. 24 has another decidedly objectionable feature, and that is the loss of finish occasioned by rubbing the sheet with the hand. The fibers under this treatment fuzz up, and considerable powder and short fiber fall off. Several of the other sheets have this same peculiarity, but if more size were added this trouble would probably be eliminated.

The experimental papers have not yet been tested on high-speed presses, and this must be done before accurate knowledge of the value of the several sheets can be had.

However, after having obtained news paper of the quality of the attached samples from hemlock, jack pine, and mixtures of these woods without changing in any way present commercial practice, it seems beyond doubt that these woods may be advantageously used either singly or in various combinations, at least in the cheaper grades of paper.

TABLE 3.—*Strength, weight, and thickness tests on experimental papers.*

Commercial run No.	Hemlock sulphite.	Spruce ground wood.	Hemlock ground wood.	Jack pine ground wood.	Weight per ream of 500 sheets 24 by 36 inches.	Average thickness.	Mullen test.		Schopper test.					
							Bursting strength per square inch.	Strength factor.	Lengthwise.			Crosswise.		
									Average stretch.	Average load on strip 18 by 1.65 cm.	Average break- ing length.	Average stretch.	Average load on strips 18 by 1.65 cm.	Average break- ing length.
	P. ct.	P. ct.	P. ct.	P. ct.	Lbs.	Inches.	Lbs.		P. ct.	Kilos.	Meters.	P. ct.	Kilos.	Met's.
1a	5	95			32	0.0033	11	0.344	0.8	2.95	3.687	1.4	1.765	2,254
1	25	75			33	.0035	13.2	.400	.95	3.49	4.043	1.22	1.780	2,150
24	25			75	33	.0038	8.2	.249	.95	2.53	3.251	1.08	1.305	1,690
46	25	25			33	.0037	11.4	.345	1.05	3.495	3.907	1.42	1.755	1,987
50	25			75	34	.0035	10.2	.300	1.11	3.615	4.111	1.17	1.555	1,819
51	25	25		25	32	.0035	9.7	.303	.95	2.855	3.495	1.24	1.410	1,692
52	25			50	33	.004	9.9	.300	.98	3.005	3.606	1.43	1.330	1,640

All of the above are averages of 10 determinations.

SUMMARY OF DATA.

Tables 4, 5, 6, 7, 8, and 9 show compilations of the data secured during tests on hemlock, jack pine, and mixtures of these two woods with spruce. The results of tests under many different conditions of speed, pressure, and surface of stone are given. In a number of cases the data on production and power consumption do not agree with that taken at another time and under the same conditions of pressure,

speed, and type of burr. In all of these instances the differing values can be accounted for by the fact that although the same kind of burr was used, the stones were of different sharpness.

It has been found difficult to duplicate in one test the surface of stone used in another under the same conditions and obtain the same production with the same power consumption. In fact, the production factors vary greatly over short periods as a result of the varying attention given by the grinder man. On this account the power and production data in the tables can be applied to commercial plants only approximately. If a grinder is operating under the conditions of any of the commercial tests shown in Tables 5, 6, and 7, however, the data given will closely approximate the actual working conditions.

TABLE 4.—Qualitative and quantitative tests on jack pine—Power consumption and production.

Kind of stone.	Run number.	Kind of burr.	Pressure on 14-inch cylinder.		Revolutions per minute.	Peripheral speed per minute.	Average horsepower to grinder.	Bone-dry pulp in 24 hours.	Horsepower per ton bone-dry pulp in 24 hours.	Bone-dry pulp per 100 cubic feet bone-dry wood.	Weight per cubic foot bone-dry wood.	Average temperature of grinding.	Size of screen slots.
			Lbs.	Lbs.		Feet.		Tons.		Lbs.	Lbs.	° F.	Inch.
Manufacturers'.	1	Natural surface.	20.0	8.20	151.0	2,100						140.0	(1)
Do.....	2		50.0	12.30	151.0	2,100						147.0	(1)
Do.....	3		39.0	16.00	150.0	2,090						176.0	(1)
Do.....	3-1		39.3	16.10	151.0	2,100						176.0	(1)
Do.....	4		50.0	20.56	150.6	2,100						196.0	(1)
Do.....	5		60.0	24.65	151.0	2,100						199.0	(1)
Do.....	6		60.0	24.65	171.0	2,380						196.0	(1)
Do.....	6-1		60.0	24.65	171.0	2,370						201.0	(1)
Do.....	7		50.0	20.50	171.0	2,370						199.0	(1)
Do.....	7-1		50.0	20.50	172.0	2,380	256.0	1.305	196.2	1,820	25.27	197.0	0.012
Do.....	7-2		50.0	20.50	172.0	2,380	245.0	1.263	193.6	2,150	25.57	183.0	.012
Do.....	8		60.0	24.65	203.0	2,810						198.0	(1)
Do.....	8-1		60.0	24.65	203.0	2,810						191.0	(1)
Do.....	9		50.0	20.50	202.0	2,800						205.0	(1)
Do.....	10		75.0	30.80	202.0	2,800						185.0	(1)
Do.....	11	Diamond point, 6 to the inch.	60.0	24.65	152.5	2,100	380.5					148.0	.012
Do.....	11-1		60.0	24.65	154.0	2,100	360.5	6.075	59.3			146.0	.012
Do.....	12		50.0	20.50	152.5	2,100	350.0	5.225	67.0			145.0	.012
Do.....	12-1		50.0	20.50	154.0	2,100	359.0	7.030	51.0			179.0	.012
Do.....	13		40.0	16.40	152.5	2,100	260.0	3.028	85.9			138.2	.012
Do.....	13-1		40.0	16.40	153.5	2,100	299.0	2.670	112.0			195.0	.012
Do.....	14		50.0	20.50	173.0	2,380	324.0	4.716	68.8			154.0	.012
Do.....	14-1		50.0	20.50	174.0	2,380	398.0	5.046	78.8			166.0	.012
Do.....	15		60.0	24.65	173.0	2,380	395.5	6.390	61.8			138.0	.012
Do.....	15-1		60.0	24.65	174.0	2,380	461.0	5.500	83.8			158.0	.012
Do.....	16		40.0	16.40	203.5	2,800	362.5	5.450	66.4			147.0	.012
Do.....	16-1		40.0	16.40	205.0	2,800	375.0	5.360	70.0			200.0	.012
Do.....	17		40.0	16.40	173.0	2,380	289.0	3.834	75.5			143.0	.012
Do.....	17-1		40.0	16.40	174.0	2,380	332.0	4.350	76.3			178.0	.012
Do.....	18		50.0	20.50	204.0	2,800	407.5	6.910	59.0			143.0	.012
Do.....	18-1		50.0	20.50	205.0	2,800	414.0	6.360	65.0			193.0	.012
Do.....	19		30.0	12.30	152.5	2,100	201.0	2.045	98.3			149.0	.012
Do.....	20		30.0	12.30	205.0	2,800	313.0	5.320	58.8			178.0	.012
Do.....	20-1		30.0	12.30	205.0	2,800	304.0	3.620	83.8	2,370	24.90	198.0	.012
Do.....	21		30.0	12.30	174.0	2,380	269.5	4.810	56.0			172.0	.012
Do.....	22		60.0	24.64	205.0	2,800	481.0	7.320	65.8			185.0	.012
Do.....	23		40.0	16.40	205.0	2,800	249.0	4.110	84.9			206.0	.012
Lombard	24		40.0	16.40	175.0	2,445	395.0	4.305	91.8	2,220	25.60	176.3	.012
			45.0	18.46									

1 Unscreened.

TABLE 5.—Qualitative and quantitative tests on hemlock—Power consumption and production.

Kind of stone.	Run number.	Kind of burr.	Pressure on 14-inch cylinder.		Pressure per square inch, pocket area.	Revolutions per minute.	Peripheral speed per minute.	Average horsepower to grinder.	Bone-dry pulp in 24 hours.	Horsepower per ton bone-dry pulp in 24 hours.	Bone-dry pulp per 100 cubic feet bone-dry wood.	Weight per cubic foot bone-dry wood.	Average temperature of grinding.	Size of screen slots.
			Lbs.	Lbs.										
Manufacturers'.	0	Straight cut, 10 to the inch.	50	20.50	170.0	2,380	288.0						131.0	(1)
Do.....	1	do.....	30	12.30	100.0	1,400	154.0						126.0	(1)
Do.....	2	do.....	30	12.30	150.0	2,100	202.0						143.0	(2)
Do.....	3	do.....	30	12.30	200.0	2,800	275.0						150.0	(2)
Do.....	4	do.....	30	12.30	225.0	3,150	301.0						155.0	(2)
Do.....	5	do.....	20	8.20	150.0	2,100	169.5	1.011	167.7				159.0	(2)
Do.....	6	do.....	20	8.20	175.0	2,450	210.0	1.380	152.0				170.0	(2)
Do.....	7	do.....	20	8.20	225.0	3,150	223.0	1.295	172.0				169.0	(2)
Do.....	2-1	do.....	30	12.30	150.0	2,100	254.0	1.965	129.0				167.0	(2)
Do.....	8	do.....	40	16.40	175.0	2,450	328.0	3.055	107.3				166.0	(2)
Do.....	9	do.....	60	24.65	175.0	2,450	430.0	4.730	91.0				168.0	(2)
Do.....	10	Spiral cut, 10 to the inch.	20	8.20	100.0	1,393	87.3						129.0	(1)
Do.....	11	do.....	40	16.40	175.0	2,440	259.0	1.430	181.6				170.0	(2)
Do.....	12	do.....	50	20.50	225.0	3,150	368.0	2.475	149.0				178.0	(2)
Do.....	13	do.....	60	24.65	225.0	3,150	475.0	3.595	132.0				171.0	(2)
Do.....	14	do.....	50	20.50	175.0	2,440	358.0	3.395	105.6				166.0	(2)
Do.....	15	do.....	60	24.65	175.0	2,440	397.0	4.044	98.4				161.5	(2)
Do.....	16	do.....	30	12.30	175.0	2,440	243.0						148.5	(2)
Do.....	17	do.....	30	12.30	200.0	2,790	297.0						146.0	(2)
Do.....	18	do.....	30	12.30	100.0	1,390	162.8						158.0	(2)
Do.....	19	do.....	50	20.50	100.0	1,390	261.0	2.470	105.7				157.0	(2)
Do.....	20	do.....	40	16.40	200.0	2,790	377.0	3.780	99.7				162.0	(2)
Do.....	21	do.....	60	24.65	100.0	1,400	281.0	2.810	100.0				160.0	(2)
Do.....	22	do.....	50	20.50	150.0	2,090	366.0	3.815	96.0				161.5	(2)
Do.....	23	Diamond-point cut, 10 to the inch.	40	16.40	175.0	2,440	347.0	3.360	103.2				159.0	(2)
Do.....	24	do.....	50	20.50	175.0	2,440	394.0	5.065	77.7				157.0	(2)
Do.....	25	do.....	60	24.65	100.0	1,390	291.5	3.212	88.0				156.0	(2)
Do.....	26	do.....	30	12.30	200.0	2,782	315.0	2.945	107.0				163.0	(2)
Do.....	27	do.....	50	20.50	200.0	2,782	432.0	5.400	80.0				160.5	(2)
Do.....	28	do.....	60	24.65	101.0	1,408	290.0						168.0	(2)
Do.....	29	do.....	50	20.50	175.0	2,435	372.0						187.0	(1)
Do.....	30	Straight cut, 4 to the inch. } 10 to the inch }	40	16.40	175.0	2,435	319.0	3.900	81.8				145.0	(2)
Do.....	31	do.....	60	24.65	100.0	1,390	269.0	3.330	80.9				145.0	(2)
Do.....	32	do.....	60	24.65	100.0	1,390	298.0	3.120	95.5				155.0	(2)
Do.....	33	do.....	50	20.50	175.0	2,435	376.0	4.000	94.0				163.0	(2)
Do.....	34	Spiral cut, 8 to the inch.	60	24.65	100.0	1,390	297.0	4.350	68.3				135.0	(2)
Do.....	35	do.....	60	24.65	100.0	1,390	284.0	2.915	97.5				147.0	(2)
Do.....	36	do.....	60	24.65	150.0	2,090	417.0	5.130	81.4				152.0	(2)
Do.....	37	do.....	60	24.65	200.0	2,782	520.0	6.890	75.5				154.0	(2)
Do.....	38	do.....	50	20.50	84.3	1,173	212.0	2.660	79.7				154.0	(2)
Do.....	39	do.....	60	24.65	85.0	1,183	271.0	2.955	91.8				150.0	(2)
Do.....	40	do.....	50	20.50	100.0	1,390	285.0	3.025	94.3				156.0	(2)
Do.....	41	do.....	50	20.50	150.0	2,090	379.0	4.450	85.2				155.0	(2)
Do.....	42	do.....	50	20.50	175.0	2,435	429.0	5.295	81.1				155.0	(2)
Do.....	43	do.....	50	20.50	200.0	2,782	439.0	5.425	80.9				145.0	(2)
Do.....	44	do.....	40	16.40	175.0	2,435	359.0	4.225	85.0				158.0	(2)
Lombard	45	Straight cut, 3 to the inch.	40	16.40	200.0	2,800	340.0	2.915	116.5				159.0	(2)
Do.....	47	do.....	55	22.60	200.0	2,800	516.0	5.370	93.2				161.0	(2)
Do.....	48	do.....	50	20.50	225.0	3,145	510.0	6.235	81.8				164.0	(2)
Do.....	50	Straight cut, 3 to the inch; spiral cut, 12 to the inch.	50	20.50	175.0	2,445	422.5	5.300	79.7	2.070	24.84		170.0	(2)

¹ Unscreened.² Size of screen slots, 0.065 and 0.012.

TABLE 6.—Commercial tests on jack pine—Power consumption and production.

GREEN MATERIAL.

Kind of stone.	Commercial run number.	Kind of burr.	Pressure on 14-inch cylinder.	Pressure per square inch, pocket area.	Revolutions per minute.	Peripheral speed per minute.	Average horsepower to grinder.	Bone-dry pulp in 24 hours.	Horsepower per ton bone-dry pulp in 24 hours.	Bone-dry pulp per 100 cubic feet bone-dry wood.	Weight per cubic foot bone-dry wood.	Average temperature of grinding.	Screenings per 100 cubic feet bone-dry wood.	Size of screen slots.
			<i>Lbs.</i>	<i>Lbs.</i>		<i>Feet.</i>		<i>Tons.</i>		<i>Lbs.</i>	<i>Lbs.</i>	<i>° F.</i>	<i>Lbs.</i>	<i>Inch.</i>
Manufacturers'.	14	Diamond-point cut, 6 to the inch.	50	20.5	169	2,389	435	5.380	80.9	2,200	24.9	153		(1)
Do.....	14	do.....	50	20.5	171	2,400	458	6.650	68.9	2,070	24.9	150		(1)
Do.....	14	do.....	50	20.5	171	2,400	430	6.850	62.7	2,130	24.9	145		(1)
Do.....	14	do.....	50	20.5	171	2,400	416	7.040	59.1		24.9	149		(1)
Do.....	14	do.....	50	20.5	171	2,400	430	6.610	65.0		24.9	149		(1)
Do.....	14	do.....	50	20.5	171	2,400	427	7.070	60.4	2,190	25.8	150		(1)
Do.....	14	do.....	50	20.5	171	2,400	447	7.550	59.2		25.8	150		(1)
Do.....	14	do.....	50	20.5	171	2,400	459	8.300	55.3		25.8	147		(1)
Do.....	14	do.....	50	20.5	171	2,400	441	7.700	57.3	2,190	25.1	147		(1)
Do.....	14	do.....	50	20.5	171	2,400	430	7.420	58.0	2,075	25.1	151		(1)
Do.....	14	do.....	50	20.5	171	2,400	421	4.960	85.0	2,025	25.1	154		(1)
Do.....	14	do.....	50	20.5	171	2,400	459	6.035	76.0	2,180	25.1	162		(1)
Do.....	14	do.....	50	20.5	171	2,400	446	6.635	67.3	2,200	25.1	150		(1)
Do.....	14	do.....	50	20.5	171	2,400	445	7.135	62.5	2,190	25.1	147		(1)
Do.....	14	do.....	50	20.5	171	2,400	462	7.440	62.2	2,160	25.4	143		(1)
Do.....	14	do.....	50	20.5	171	2,400	452	6.890	66.0	2,153	25.4	148		(1)
Do.....	14	do.....	50	20.5	171	2,400	484	7.560	64.0	2,155	25.4	142		(1)
Do.....	14	do.....	50	20.5	171	2,400	467	7.335	63.7	2,235	25.4	137		(1)
		Weighted averages.					436	7.030		2,170		148		

¹ Size of screen slots, 0.065 and 0.012.

SEASONED MATERIAL.

Manufacturers'.	14	Diamond-point, cut 6 to the inch.	50	20.5	171	2,400	457	5.140	89.0	2,125	25.4	147		{0.065 .012
Do.....	14	do.....	50	20.5	171	2,400	451	5.570	81.0	2,170	25.6	142		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	468	6.780	69.0	2,210	25.6	140		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	455	7.050	64.5	2,260	25.6	136		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	460	7.365	62.4	2,233	25.6	142		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	490	6.160	79.5	2,150	25.6	154		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	386	3.995	96.6	2,095	25.6	152		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	421	6.240	67.6	2,225	25.6	144		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	444	6.340	70.0	2,320	25.6	152		{.065 .012
Do.....	14	do.....	50	20.5	171	2,400	417	5.990	69.7		25.6	146		{.065 .012
		Weighted averages.					447	6.250	72.7	2,210		145		

SEASONED MATERIAL.

Lombard.	24	Straight cut, 3 to the inch; spiral cut, 12 to the inch.	40	16.4	175	2,445	385	4.190	91.9	2,220	25.6	169.4		{0.065 .012
Do.....	24	do.....	45	18.46	175	2,445	404	4.400	91.8	2,220	25.6	182.0		{.065 .012
		Weighted averages.					395	4.305	91.8	2,220	25.6	176.3	14.9	

TABLE 7.—Quantitative and commercial test on mixtures of spruce, jack pine, and hemlock—Power consumption and production.

Kind of stone.	Commercial run number.	Kind of burr.	Pressure on 14-inch cylinder.	Pressure per square inch, pocket area.	Revolutions per minute.	Peripheral speed per minute.	Average horsepower to grinder.	Bone-dry pulp in 24 hours.	Horsepower per ton bone-dry pulp in 24 hours.	Bone-dry pulp per 100 cubic feet bone-dry wood.	Weight per cubic foot bone-dry wood.	Average temperature of grinding.	Screenings per 100 cubic feet bone-dry wood.	Size of screen slots.
$\frac{1}{3}$ spruce; $\frac{2}{3}$ hemlock:			<i>Lbs.</i>	<i>Lbs.</i>		<i>Ft.</i>		<i>Tons.</i>		<i>Lbs.</i>	<i>Lbs.</i>	<i>° F.</i>	<i>Lbs.</i>	<i>In.</i>
Lombard.....	46A	{Straight cut, 3 to the inch.	50	20.5	175	2,450	392.0	4.275	91.7			152.5		{.065 .012
Hemlock:														
Lombard.....	46B	do.....	50	20.5	175	2,450	413.0	4.885	84.6			175.5		{.065 .012
Spruce:														
Lombard.....	46C	do.....	50	20.5	175	2,450	407.0	4.878	83.5			160.0		{.065 .012
$\frac{1}{3}$ spruce; $\frac{2}{3}$ hemlock:														
Lombard.....	49A	Straight cut, 3 to the inch; spiral cut, 12 to the inch.	40	16.4	175	2,445	360.4	4.880	73.8			155.0		{.065 .012
Hemlock:														
Lombard.....	49B	do.....	40	16.4	175	2,445	370.0	4.885	75.7			168.3		{.065 .012

COMMERCIAL RUNS.

$\frac{1}{3}$ spruce; $\frac{2}{3}$ hemlock:																
Lombard.....	46A	{Straight cut, 3 to the inch; spiral cut, 12 to the inch.	50	20.5	175	2,445	426.0	5.660	75.3			149.0		{.065 .012		
Do.....	46A		do.....	50	20.5	175	2,445	425.0	5.135	82.7			173.0			
Do.....	46A		do.....	50	20.5	175	2,445	432.0	4.905	88.0			176.3			
		Weighted averages.	}				427.2	5.175	83.0	2,030	{24.8 (23.76)	169.0	16.3			
$\frac{1}{3}$ jack pine; $\frac{1}{3}$ spruce; $\frac{1}{3}$ hemlock:																
Lombard.....	51	{Straight cut, 3 to the inch; spiral cut, 12 to the inch.	50	20.5	175	2,445	442.0	5.705	77.5			167.6		{.065 .012		
Do.....	51		do.....	50	20.5	175	2,445	444.0	5.420	81.9			175.0			
			Weighted averages.	}				443.0	5.575	79.4	2,232	{25.6 (24.7 (29.11)	171.0		17.4	
$\frac{1}{3}$ jack pine; $\frac{2}{3}$ hemlock:																
Lombard.....	52	{Straight cut, 3 to the inch; spiral cut, 12 to the inch.	50	20.5	175	2,445	398.0	4.550	87.5			160.0		{.065 .012		
Do.....	52		do.....	50	20.5	175	2,445	425.0	4.350	97.7			185.4			
Do.....	52		do.....	50	20.5	175	2,445	406.5	3.950	103.0			179.4			
		Weighted averages.	}				414.0	4.310	96.3	2,230	{25.2 (26.75)	177.7	17.6			

TABLE 8.—Commercial tests on hemlock—Power consumption and production.

Kind of stone.	Commercial run number.	Kind of burr.	Pressure on 14-inch cylinder.		Revolutions per minute.	Peripheral speed per minute.	Average horsepower to grinder.	Bone-dry pulp in 24 hours.	Horsepower per ton bone-dry pulp in 24 hours.	Bone-dry pulp per 100 cubic feet bone-dry wood.	Weight per cubic foot bone-dry wood.	Average temperature of grinding.	Screenings per 100 cubic feet bone-dry wood.	Size of screen slots.
			Lbs.	Lbs.		Ft.								
Manufacturers' ..	14	{Spiral cut, 10 to the inch.	50	20.5	175	2,435	331.0	2.855	116.0	1,955	24.8	173.5		{.065 .012
Do.....	14	do.....	50	20.5	175	2,435	356.0	3.795	94.0	2,030	24.8	168.0		{.065 .012
Do.....	14	do.....	50	20.5	175	2,435	353.0	3.425	103.0	2,122	24.8	173.0	10.6	{.065 .012
		Weighted averages.					349.0	3.417	103.1	2,048	24.8	171.4		
Manufacturers' ..	14-1	{Spiral cut, 10 to the inch.	50	20.5	175	2,432	363.0	4.195	86.5	2,070	24.8	165.5	18.6	{.065 .012
Do.....	14-1	do.....	50	20.5	175	2,432	371.5	4.068	91.4	2,080	24.8	168.0	18.5	{.065 .012
		Weighted averages.					368.2	4.115	89.5	2,077	24.8	167.0		
Manufacturers'...	8	{Straight cut, 10 to the inch.	40	16.4	176	2,450	346.0	3.945	87.7	2,118	24.8	165.5	24.8	{.065 .012
Do.....	8	do.....	40	16.4	176	2,450	287.0	3.410	84.2	2,085	24.8	159.0	15.5	{.065 .012
Do.....	8	do.....	40	16.4	176	2,450	270.0	2.772	97.5	2,038	24.8	177.6	18.6	{.065 .012
		Weighted averages.					306.0	3.435	89.7	2,083	24.8	168.4		
Manufacturers'...	23	{Diamond point, cut 10 to the inch.	40	16.4	176	2,440	300.0	2.740	109.5	2,040	24.8	186.5	13.7	{.065 .012
Do.....	23	do.....	40	16.4	176	2,440	318.0	3.635	87.5	2,125	24.8	172.0	32.7	{.065 .012
Do.....	23	do.....	40	16.4	176	2,432	281.0	3.350	83.9	2,130	24.8	162.0	20.3	{.065 .012
		Weighted averages.					301.0	3.300	92.4	2,105	24.8	172.8		
Lombard.....	30	{Straight cut, 4 to the inch; spiral cut, 10 to the inch.	40	16.4	176	2,432	340.0	4.075	83.5	2,080	24.8	176.0	16.5	{.065 .012
Do.....	30	do.....	40	16.4	176	2,432	315.0	3.170	99.4	2,140	24.8	179.5	17.8	{.065 .012
		Weighted averages.					331.0	3.725	89.5	2,102	24.8	177.0		
Lombard.....	50	{Straight cut, 3 to the inch; spiral cut, 12 to the inch.	50	20.5	175	2,445	417.0	4.05	103.0	2,195	25.2	184.5	11.2	{.065 .012
Do.....	50	do.....	50	20.5	175	2,445	417.0	3.815	109.2	2,195	25.2	185.0	11.2	{.065 .012
Do.....	50	do.....	50	20.5	175	2,445	422.5	5.300	79.7	2,070	24.84	170.6		{.065 .012
		Weighted averages.					418.0	4.370	97.5	2,160	25.1	180.3		

TABLE 9.—Quantitative and commercial tests on spruce—Power consumption and production.

Kind of stone.	Run number.	Kind of burr.	Pressure on 14-inch cylinder.	Pressure per square inch pocket area.	Revolutions per minute.	Peripheral speed per minute.	Average horsepower to grinder.	Bone-dry pulp in 24 hours.	Horsepower per ton bone-dry pulp in 24 hours.	Bone-dry pulp per 100 cubic feet bone-dry wood.	Weight per cubic foot bone-dry wood.	Average temperature of grinding.	Screenings per 100 cubic feet bone-dry wood.	Size of screen slots.
			Lbs.	Lbs.		Ft.		Tons.		Lbs.	Lbs.	° F.	Lbs.	In.
Lombard ..	1	Straight cut, 3 to the inch; spiral cut, 12 to the inch.	40 50	16.4 20.5	175	2,445	403	4.988	80.8			171.0		{0.065 0.012
Do.....	1	do	40 50	16.4 20.5	175	2,445	399	4.525	88.1			175.5		{0.065 0.012
		Weighted averages.					401	4.810	83.5	2,480	28.4	172.6	17.2	
Do.....	2	Straight cut, 3 to the inch; spiral cut, 12 to the inch.	40	16.4	200	2,795	398	4.245	93.8			164.3		
Do.....	2	do	40	16.4	200	2,795	408	3.995	102.0			171.3		
Do.....	2	do	40	16.4	200	2,795	394	4.175	94.4			166.7		
		Weighted averages.					402	4.120	97.5	2,012	22.72	168.0	13.75	
	3	Straight cut, 3 to the inch; spiral cut, 12 to the inch.	20	8.2	175	2,445	191	1.215	157.0	2,300	27.66	163.0	9.82	
	4	do	40	16.4	175	2,445	333	3.025	110.0	2,408	27.66	166.5	17.8	
	5	do	60	24.65	175	2,445	454	5.255	86.4	2,415	27.66	152.2	18.6	

TABLE 10.—Furnish to beater on basis of 1,000 pounds of stock—Experimental and commercial papers.

EXPERIMENTAL PAPER.

Run.	Number.	Hemlock sulphite.	Spruce ground wood.	Hemlock ground wood.	Jack pine ground wood.	Size.	Alum.	Clay.	Rhodamine B. extra.	Soluble blue H. A.	Diamond green C. N.	Auromine O. O. D.	Orange R. O.
		Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Oz.	Oz.	Oz.	Oz.	Oz.
Commercial.....	1A	50	950			8	8	20	0.067	3.33			
Do.....	1	250	750			8	8	20	.067	3.33			
Do.....	24	250			750	8	8	20		3.6	0.15		
Do.....	46	250	250	500		8	8	20		4.0	.15		
Do.....	50	250		750		8	8	20		3.7	.20		
Do.....	51	250	250	250	250	8	8	20		4.1	.15		
Do.....	52	250		500	250	8	8	20		4.3	.15		

COMMERCIAL PAPER.

No. 2 white manila.....	210	395	395		8	8		2.8				
Butchers manila.....	210	263	527		20	20	26.7				2.75	0.83



Paper Sample from Run No. 1

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock).

Spruce Ground-wood (Run No. 1)		750	lbs.
Hemlock Quick-cook Sulphite		250	"
Size		8	"
Alum		8	"
Clay		20	"
Color		—	
Rhodamine B. extra		0.067	oz.
Soluble Blue H. A.		3.33	"

SECTION OF
RY
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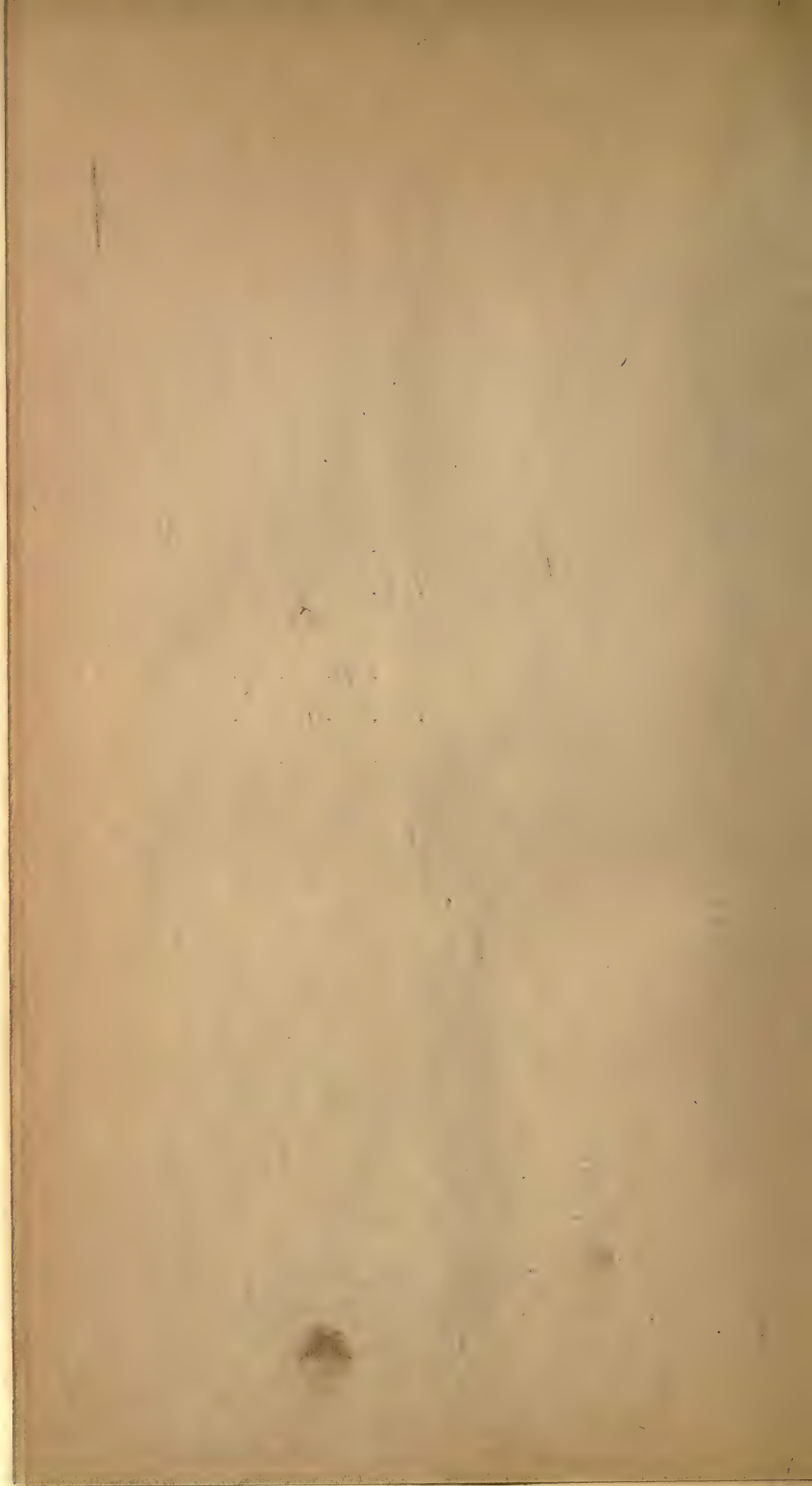
Paper Sample from Run No. 1A

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Spruce Ground-wood (Run No. 1)	.	.	.	.	950	lbs.
Hemlock Quick-cook Sulphite	.	.	.	.	50	"
Size	.	.	.	.	8	"
Alum	.	.	.	.	8	"
Clay	.	.	.	.	20	"
Color	.	.	.	.	—	
Rhodamine B. extra	.	.	.	.	0.067	oz.
Soluble Blue H. A	.	.	.	.	3.33	"

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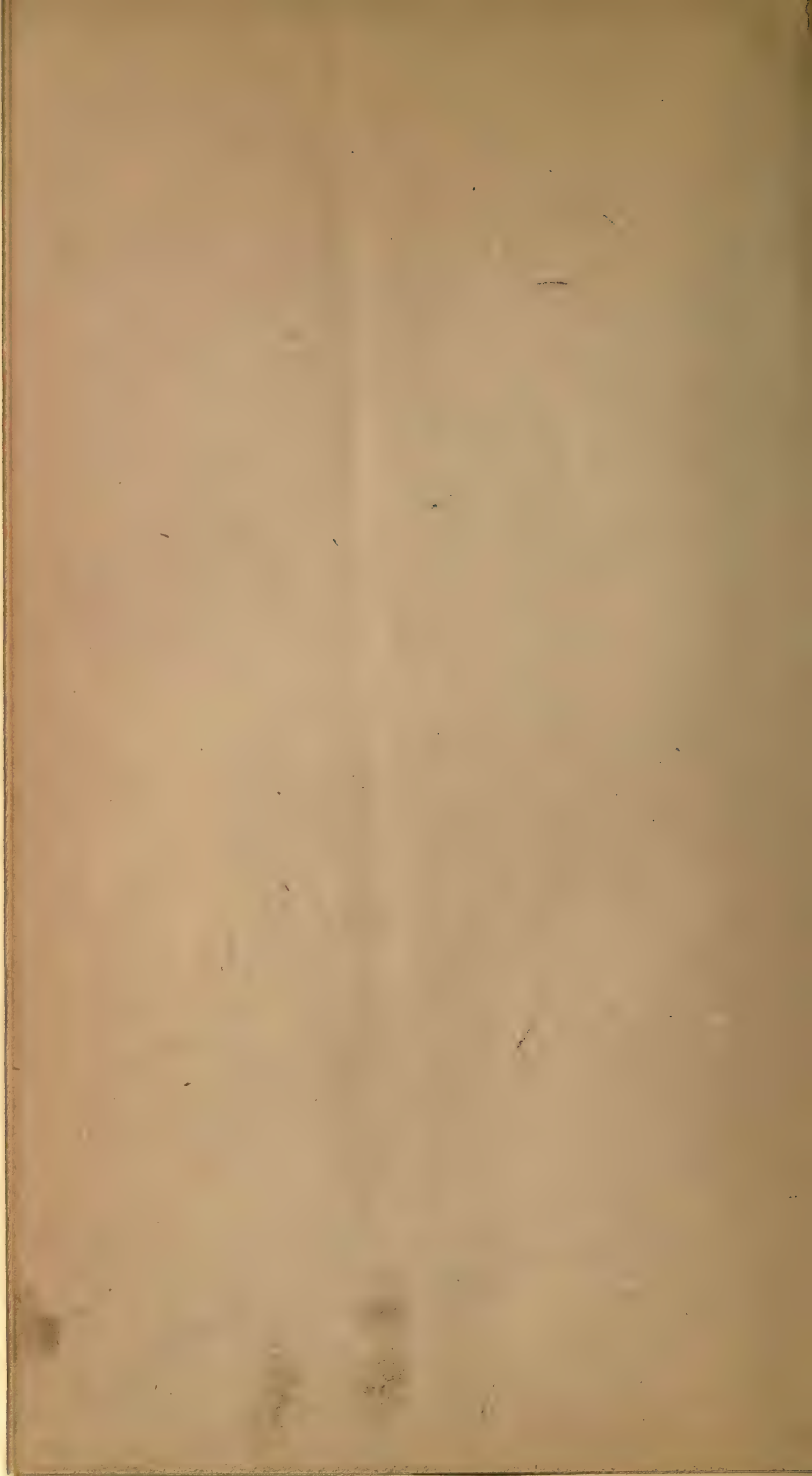
Paper Sample, No. 2 White Manila

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Spruce Ground-wood	395	lbs.
Hemlock Ground-wood	395	"
Hemlock Quick-cook Sulphite	210	"
Size	8	"
Alum	8	"
Clay	—	
Color	—	
Soluble Blue H. A.	2.8	oz.

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Paper Sample, Butcher's Manila

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Spruce Ground-wood	263	lbs.
Hemlock Ground-wood	527	"
Hemlock Quick-cook Sulphite	210	"
Size	20	"
Alum	20	"
Clay	26.7	"
Color	—	
Auromine O. O. D.	2.75	oz.
Orange R. O.	0.83	"

VISION OF

TRY

CULTURE
TORNIA

CHURCH & PARSONS, MIDDLETOWN, CT

March 1st

Received of Mr. J. H. Smith

for balance of account

the sum of \$100.00

and

for

rent

of \$10.00

for commission

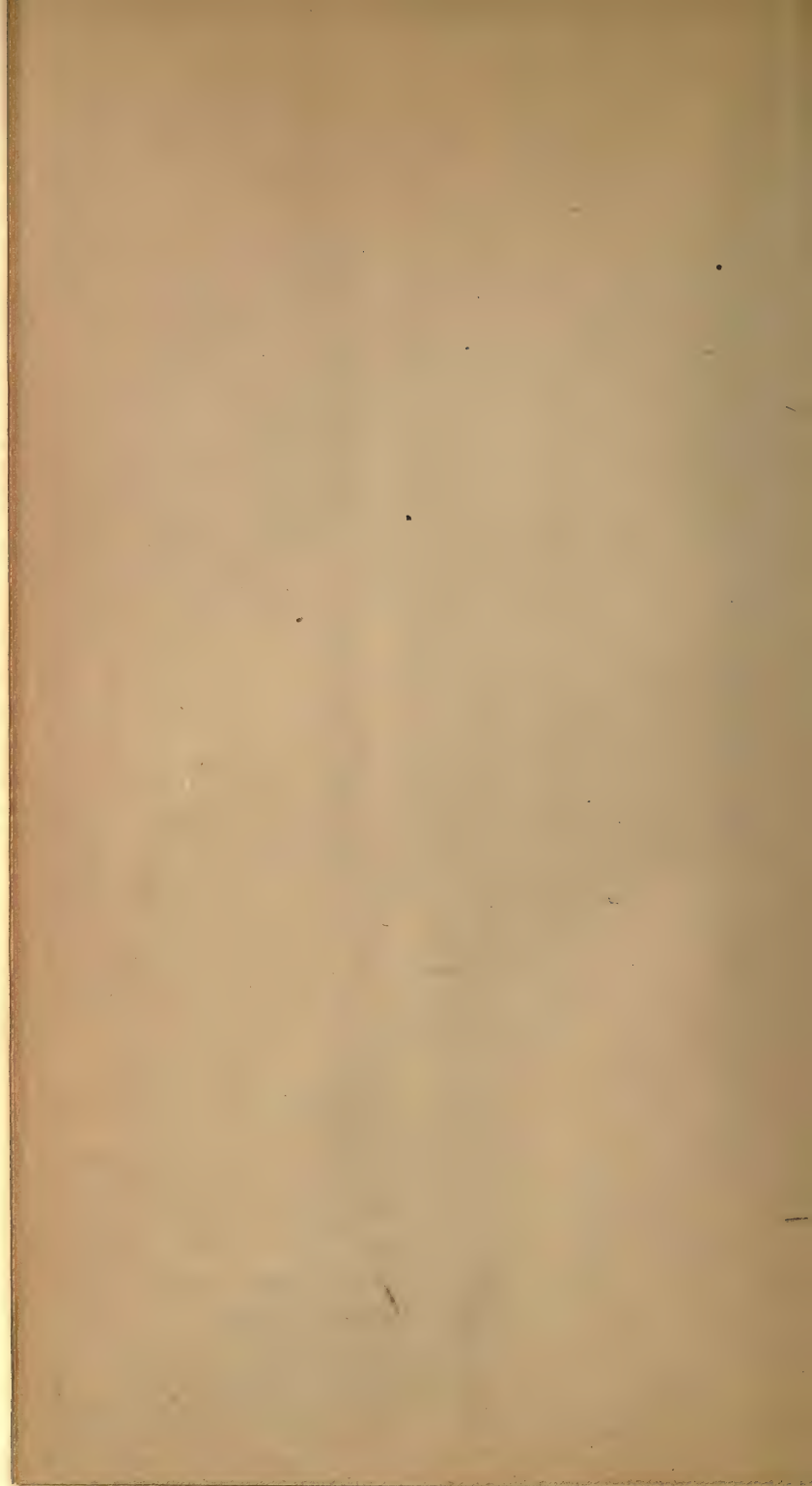
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Paper Sample from Run No. 24

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Jack Pine Ground-wood (Run No. 24)	. . .	750	lbs.
Hemlock Quick-cook Sulphite	. . .	250	"
Size	. . .	8	"
Alum	. . .	8	"
Clay	. . .	20	"
Color	. . .	—	
Soluble Blue H. A.	. . .	3.6	oz.
Diamond Green G. N.	. . .	0.15	"



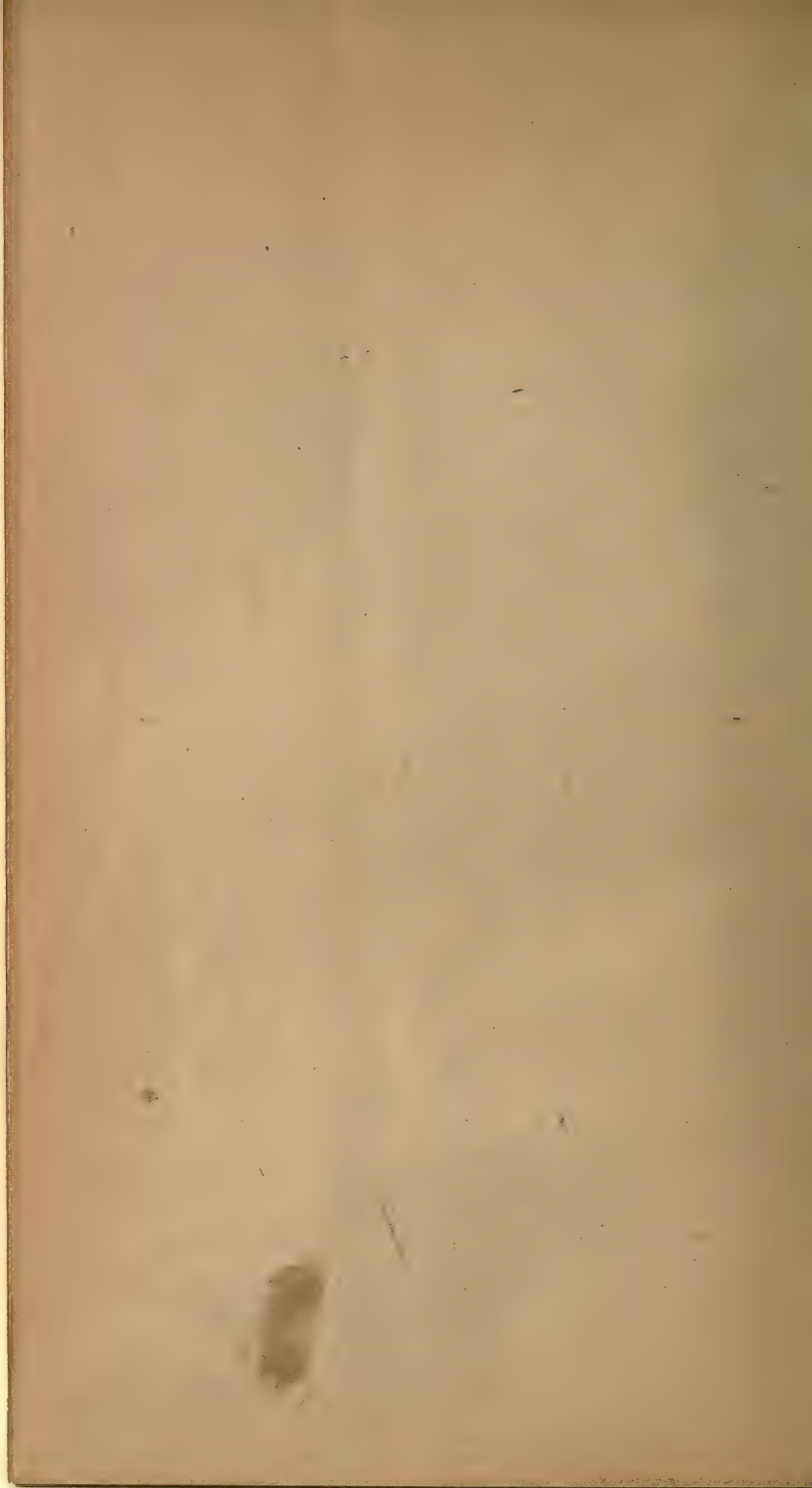
Paper Sample from Run No. 46

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Spruce Ground-wood (Run No. 46)		250	lbs.
Hemlock Ground-wood (Run No. 46)		500	"
Hemlock Quick-cook Sulphite		250	"
Size		8	"
Alum		8	"
Clay		20	
Color		—	
Soluble Blue H. A.		4	oz.
Diamond Green G. N.		0.15	"

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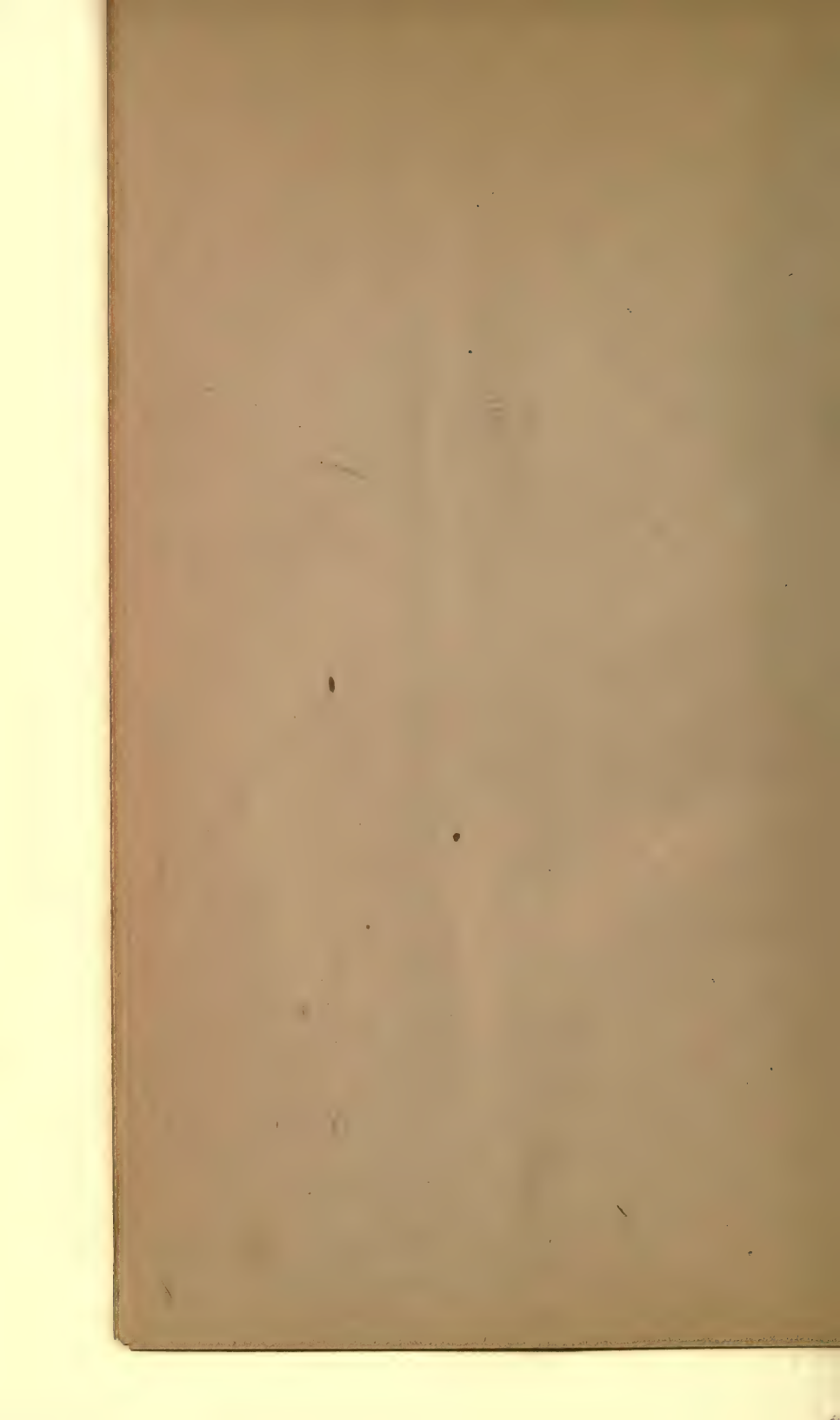
Paper Sample from Run No. 50

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Hemlock Ground-wood (Run No. 50)	750	lbs.
Hemlock Quick-cook Sulphite	250	"
Size	8	"
Alum	8	"
Clay	20	"
Color	—	
Soluble Blue H. A.	3.7	oz.
Diamond Green G. N.	0.20	"

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Paper Sample from Run No. 51

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Spruce Ground-wood (Run No. 51)		250	lbs.
Hemlock Ground-wood (Run No. 51)		250	"
Jack Pine Ground-wood (Run No. 51)		250	"
Hemlock Quick-cook Sulphite		250	"
Size		8	"
Alum		8	"
Clay		20	"
Color		—	
Soluble Blue H. A.		4.1	oz.
Diamond Green G. N.		0.15	"

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Paper Sample from Run No. 52

Furnish

(Figured on basis of 1,000 pounds of bone-dry stock.)

Hemlock Ground-wood (Run No. 52)	. . .	500	lbs.
Jack Pine Ground-wood (Run No. 52)	. . .	250	"
Hemlock Quick-cook Sulphite	. . .	250	"
Size	. . .	8	"
Alum	. . .	8	"
Clay	. . .	20	"
Color	. . .	—	
Soluble Blue H. A.	. . .	4.3	oz.
Diamond Green G. N.	. . .	0.15	"

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Issued January 11, 1913.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE—BULLETIN 105.

HENRY S. GRAVES, Forester.

FOREST PRODUCTS LABORATORY SERIES.

WOOD TURPENTINES:

THEIR ANALYSIS, REFINING, AND COMPOSITION,
BASED UPON EXPERIMENTS AT THE FOREST
PRODUCTS LABORATORY AT MADISON, WIS.

BY

L. F. HAWLEY,

Chemist in Forest Products.

PROPERTY OF DIVISION OF

FORESTRY
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1913.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE,

Washington, D. C., January 30, 1912.

SIR: I have the honor to transmit herewith a manuscript entitled "Wood Turpentine: Their Analysis, Refining, and Composition," by L. F. Hawley, chemist in forest products, and to recommend its publication as Bulletin 105 of the Forest Service. The investigation was carried on at the Forest Products Laboratory maintained in cooperation with the University of Wisconsin. The Yellow Pine Manufacturers' Association contributed funds toward the purchase of the refining still. The crude turpentines used in the refining experiments were obtained through cooperation with the producers. Mr. Ernest Bateman, chemist in forest products, collaborated in the development of the analytical methods and analyzed some of the products of the refining. Mr. F. E. Lichtenthaler, formerly assistant chemist, did most of the work in connection with the installation of the refining apparatus.

Respectfully,

HENRY S. GRAVES,
Forester.

HON. JAMES WILSON,
Secretary of Agriculture.

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WOOD TURPENTINES: THEIR ANALYSIS, REFINING, AND COMPOSITION.

PURPOSE OF THE STUDY.

Crude wood turpentine is one of the important products obtained by most of the chemical industries which use waste resinous wood as a raw material. Refined turpentine from these crude wood distillates have been on the market for several years, but there has been a general lack of authentic information concerning them, especially on the relations existing between methods of production, their composition, and their properties. No simple method existed for the determination of differences in their composition. Moreover, strong prejudice has developed against any product sold under the name of wood turpentine, which has never been standardized, so that there exists a large variation in the products from different plants. Also there is a marked difference in odor between most of the wood turpentine and the commonly used "gum" turpentine or "spirits," and still further this prejudice has been aided by a general lack of definite information. Naturally this has resulted in a poor market for wood turpentine, with a correspondingly slow development of the industry and incomplete utilization of the various kinds of waste wood available. It was for the purpose of improving these conditions that this investigation was undertaken.

The following results have been obtained:

(1) A simple distillation method has been developed for the examination of turpentine. From it more complete and accurate conclusions can be drawn in regard to the composition of a turpentine than could be had from former methods of equal simplicity.

(2) Methods of refining applicable to wood turpentine of a wide range in composition have been described, based on the refining of five samples of crude wood turpentine produced by as many different commercial processes.

(3) It has been shown how the composition of wood turpentine is influenced by different methods of production, by refining, and by storage, and how wood turpentine compare with gum turpentine in composition.

The next step in the investigation, the study of the relations between composition and technical value, has been begun, but it will be some time before the results are complete.

METHOD OF ANALYSIS.

It was necessary to devise a method for the examination of the crude turpentine and the volatile products obtained from them which would fulfill the following requirements:

- (1) Show quantitatively the proportions of the main constituents of the material examined, especially of those constituents which were to be separated in the refining process.
- (2) Show every possible difference in composition between the various refined turpentine.
- (3) Be fairly simple and rapid in operation.

No method was available which fulfilled all these conditions. The ordinary methods for the examination of turpentine apply only to the refined product (usually to gum turpentine) and are intended mainly for the detection of adulterations. The method described in Forest Service Circular 152¹ most nearly fulfills these conditions, because instead of determining the properties of the turpentine as a whole the turpentine is first fractionated by distillation with steam and the properties of the fractions are determined. By this method some idea of the proportion of the various constituents can be obtained.

After an analysis by this method, however, the composition of the turpentine could be expressed only in terms of "constituents uniform in composition" and "heavy residue" without the possibility of distinguishing between the different kinds of turpentine or of obtaining indications of the actual chemical constituents present. It was found also (see pp. — to —) that the separation of the main constituents of the turpentine by this method of distillation was so incomplete—especially in cases where comparatively large amounts of "heavy residue" were present—that the quantitative expression of the composition of the turpentine, even in such general terms as "constituents uniform in composition" and "heavy residue," was not accurate. It was necessary, therefore, to develop and adapt for use in this investigation another method for the examination of turpentine.

DESCRIPTION OF METHOD.

This new method is similar in general principles to that described in Circular 152, but it is modified in several details to make it simpler, more accurate, and more rapid.

¹ "The Analysis of Turpentine by Fractional Distillation with Steam," by W. C. Geer.

FRACTIONAL DISTILLATION.

Five hundred grams of the turpentine to be analyzed are weighed in a round-bottomed flask of about 1,000 cubic centimeter capacity; the flask is connected with a condenser by a Hempel column of the shape and size shown in figure 1, in the top of which is placed a thermometer with the bulb reaching to within 1 inch of the beads. The flask is heated on a sand bath or a piece of asbestos board and, in order to keep the liquid boiling smoothly, without bumping, a small flat coil of copper or nickeled wire is laid on the bottom of the flask. If the room temperature is very low a shield of asbestos board around the flask may also be desirable. The distillation is carried on at a maximum rate of two drops a second¹ and the distillate is caught in weighed flasks of about 100 c. c. capacity.

Neither the size of the fractions—that is, the amount of distillate to be caught in each separate flask—nor the amount of undistilled residue to be left in the distilling flask can be definitely specified, since the proper amounts may vary with different kinds of turpentines. In general, however, the fractions may well be larger (10 to 12 per cent) when the temperature of the distillation is changing slowly and regularly, but should be smaller (4 to 5 per cent) when the temperature is changing rapidly. Usually, also, it is unnecessary to continue the distillation after a temperature of 180° C. (corrected) is reached, because this seems to be the temperature below which almost all of the turpentine materials are distilled over and above which the heavy oils begin to come over in large quantities. This is especially true in the case of wood turpentines. With gum turpentines and some wood turpentines, however, especially those with a small amount of heavy oil, the turpentine materials are all distilled over before this temperature is attained.

There are many details of manipulation which can not be completely specified in a method which is to be applied to materials of

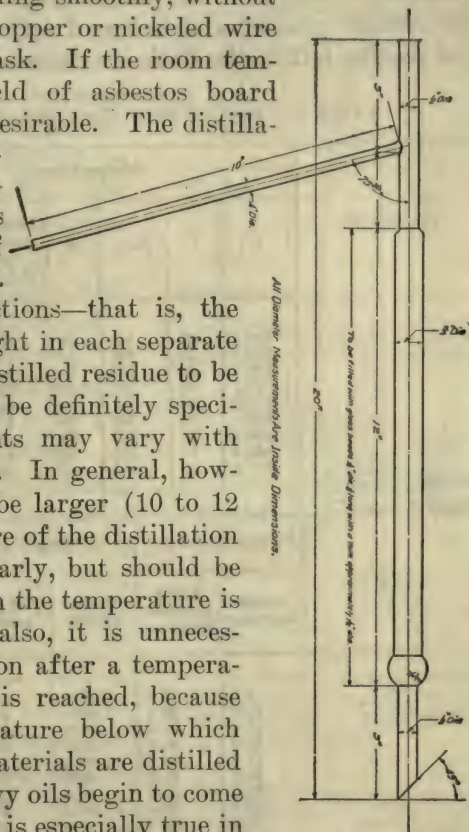


FIG. 1.—Hempel column.

¹ A slower distillation gives, of course, a better fractionation, and the maximum rate mentioned should never be exceeded; there should, moreover, be no tendency to hasten the distillation in order to save time, because the work of determining the amount and properties of the fractions can be carried out at the same time as the distillation. Indeed, a moderately rapid and experienced worker, with all the apparatus handy, can make the necessary determinations and computations, and can record and plot the results on six or seven fractions, while nine or ten are being distilled.

widely variable character. However, ordinary intelligence and a little experience are sufficient to indicate the proper variations in detail.

EXAMINATION OF FRACTIONS.

The weight of each fraction is determined and the percentage weights of each fraction and of the total combined fractions (see Table 1) are then computed. The maximum boiling point of each fraction is read from the thermometer at the top of the column, and the specific gravity and index of refraction of each fraction and of the residue left in the flask are determined.

TABLE 1.—*Typical data sheet showing records of distillations.*

Fraction No.	Temperature.						Weight.		
	<i>T.</i>	<i>t.</i>	<i>T-t.</i>	<i>N.</i>	Corrected barometer, 741.5 mm.	Corrected barometer, 760 mm.	Flask plus distillate.	Flask.	Distillate.
							grams.	grams.	grams.
1.....	156.5	49	108	56	157.4	158.5	79.6	32.1	47.5
2.....	157.0	50	107	57	157.9	159.0	94.1	39.0	55.1
3.....	157.3	50	107	57	158.2	159.3	80.3	25.3	55.0
4.....	157.7	51	107	57	158.6	159.7	97.6	36.6	61.0
5.....	158.0	50	108	58	158.9	160.0	83.1	28.3	54.8
6.....	158.8	50	109	58	159.7	160.8	96.7	32.5	64.2
7.....	159.7	51	109	59	160.6	161.7	77.4	27.3	50.1
8.....	162.4	51	111	62	163.4	164.5	96.2	43.5	52.7
9.....	167.3	52	115	67	168.4	169.5	61.0	27.1	33.9
Residue.....							150.6	125.5	25.1

Fraction No.	Percentage weight.		Specific gravity.			Index of refraction.		
	Single fractions.	Total combined fractions.	Determined.	Temperature.	Calculated for 15° C.	Determined.	Temperature.	Calculated for 15° C.
				°C.			°C.	
1.....	9.5	9.5	0.8596	21.5	0.8650	1.4693	19.5	1.4714
2.....	11.0	20.5	.8597	21.5	.8651	1.4694	19.5	1.4715
3.....	11.0	31.5	.8599	21.5	.8653	1.4695	19.5	1.4716
4.....	12.2	43.7	.8601	21.5	.8655	1.4699	19.5	1.4720
5.....	11.0	54.7	.8605	21.2	.8656	1.4702	19.5	1.4723
6.....	12.8	67.5	.8609	21.2	.8660	1.4707	19.2	1.4727
7.....	10.0	77.5	.8615	21.0	.8665	1.4714	19.2	1.4734
8.....	10.5	88.0	.8625	21.0	.8675	1.4730	19.2	1.4750
9.....	6.8	94.8	.8639	21.0	.8689	1.4760	19.2	1.4780
Residue.....	5.0	99.8	.9408	20.0	.9450	1.4914	21.1	1.4943

Boiling point.—The boiling temperatures as read should be corrected (1) for the prevailing barometric pressure, using the factor 0.056° for every millimeter difference from the normal, 760 mm., and (2) for the emergent stem of the thermometer according to the following formula:¹ To the observed value of the boiling point, *T*, should be added the factor 0.000143 (*T-t*) *N*, in which *t* is the temperature of the thermometer stem above the cork and *N* the length expressed in degrees of the mercury column above the cork.

¹ Young's "Fractional Distillation," p. 12.

Specific gravity.—The specific gravity can be determined accurately enough by means of a Westphal balance, making the determinations at room temperature and correcting to 15° C. by using the factor 0.00083 for every degree centigrade of difference from this standard temperature. The correction for temperature is large enough so that especial care must be taken to make the temperature readings accurate. In order to make specific gravity determinations with the Westphal balance on as small a fraction as is often desirable, it is necessary to have a smaller cell than that usually furnished with the instrument. This can readily be made by properly shortening a test tube with a diameter only slightly greater than the bob of the balance and supporting it in a flat cork. In this way the gravity of a fraction of only 18 grams (3.6 per cent) can readily be determined on a balance with a bob having a displacement of 5 c. c.

Index of refraction.—The index of refraction can be determined with sufficient accuracy by means of an Abbé refractometer, taking the readings at room temperature and correcting by means of the factor 0.00047 for every degree difference from the standard temperature of 15° C. It has been found that this factor 0.00047 is more nearly correct for the general run of turpentines than 0.00049 (as recommended in Circular No. 152) and that the use of different temperature correction factors for different values of the index of refraction is not necessary in order to obtain the degree of accuracy required for the work.

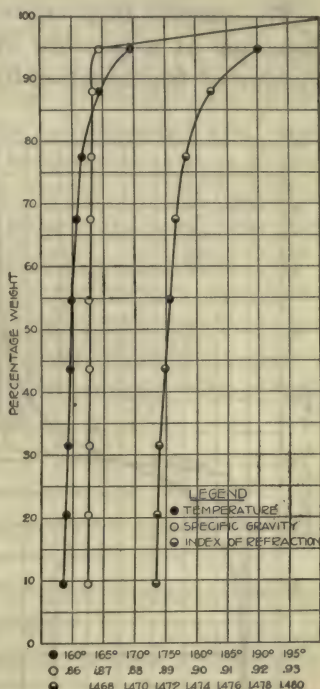


FIG. 2.—Typical curves showing boiling points, specific gravities, and indices of refraction of a gum turpentine.

RECORDING DATA.

A typical data sheet showing the original and corrected values as first recorded is shown in Table 1. For the purpose of making the interpretation of these results simpler and easier the final corrected values for the boiling point, specific gravity, and index of refraction of the different fractions are plotted against the total percentage weight of these fractions. The curves resulting from plotting the data given in Table 1 are shown in figure 2. In other distillations

the original and corrected data will not be given, but only the curves obtained by plotting the corrected data; the curves are sufficient to give the complete interpretation of the results, and there are so many distillations recorded that it would be prohibitive to use the space required by the tabulated data.

INTERPRETATION OF DISTILLATION CURVES.

To interpret these curves properly it is necessary to understand the behavior of a mixture of liquids on distillation, and a brief discussion of fractionation by distillation and of distillation curves will therefore be given, to be followed by a discussion of the curves obtained by the distillation of two different turpentines.

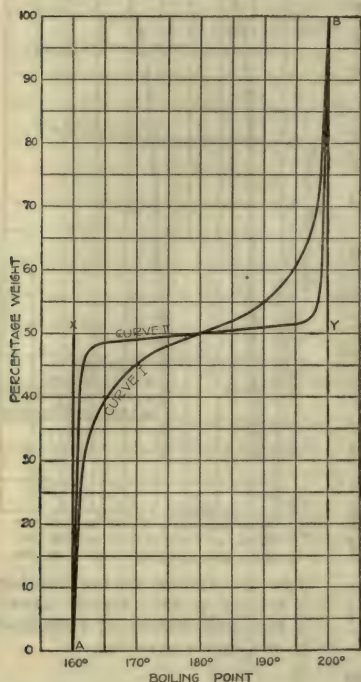


FIG. 3.—Curves illustrating theory of fractional distillation.

ways affected by the presence of another volatile liquid. In this case some of the higher boiling substance B would distill over with A long before the temperature of the distillation reaches 200°, the boiling point of B, and some of the lower boiling substance A would be left in the flask even after the temperature had risen above 160° C. When such a mixture is distilled from an ordinary distilling flask, the distillation curve would therefore be more like curve I in figure 3. This curve shows that the composition of the distillate gradually changes throughout the distillation from pure A to pure B, the first

THEORETICAL BOILING-POINT CURVES.

Let us assume a mixture of equal quantities of two volatile liquids, A and B, with boiling points 160° C. and 200° C., respectively. If on distilling this mixture a complete separation of the two components was obtained, then the distillation curves formed by plotting the percentage of distillate against the boiling point would be represented by the vertical lines AX and BY (fig. 3); that is, all of substance A in pure condition would distill over first and then the pure substance B.

FRACTIONATION BY ORDINARY DISTILLATION.

Such a complete separation is, however, never obtained, because the boiling point of one liquid is always affected by the presence of another volatile liquid. In this case some of the higher boiling substance B would distill over with A long before the temperature of the distillation reaches 200°, the boiling point of B, and some of the lower boiling substance A would be left in the flask even after the temperature had risen above 160° C. When such a mixture is distilled from an ordinary distilling flask, the distillation curve would therefore be more like curve I in figure 3. This curve shows that the composition of the distillate gradually changes throughout the distillation from pure A to pure B, the first

25 per cent of the distillate being nearly pure A and the last 25 per cent being nearly pure B.

FRACTIONATION BY DEPHLEGMATION.

In order to obtain better separations of A from B in a single distillation some kind of a still head or dephlegmator is used (such as the Hempel column shown in fig. 1) in which the ascending vapors are partially condensed and flow downward in liquid form to the distilling flask. The descending liquid comes in contact with more ascending vapors, and there is an interchange of constituents, some of the low boiling material in the liquid being vaporized and some of the high boiling material in the vapors being condensed, with the result that the vapors which finally leave the top of the dephlegmator contain a larger proportion of the lower boiling material than the vapors which enter the dephlegmator from the flask. In other words, when the dephlegmator is used there is a more complete separation of the component materials. Under such conditions the distillation curve would be more like curve II in figure 3, which indicates about 45 per cent practically pure A, about 10 per cent of a rapidly changing mixture, and the rest practically pure B.

SUMMARY.

This consideration of the boiling-point curve of a mixture of two components may be summed up as follows:

(1) The more complete the separation the more accurately do the curves represent the composition of the mixture.

(2) The more vertical curves indicate a more uniform substance in the distillate.

(3) Curves more nearly horizontal indicate a distillate rapidly changing in composition.

DISTILLATION CURVES OF A GUM TURPENTINE.

There is an analogy between the distillation curves for a turpentine and the theoretical curve in figure 3, but complications arise in the interpretation because the mixture distilled is complex and the separation of some of the constituents is incomplete. Specific gravity and index of refraction are plotted, like the boiling point, against percentage weight, and the three curves are considered together in interpreting the results of the distillation.

The curves in figure 2, which give the results of the distillation of a gum turpentine, might be interpreted, in the light of the previous discussion, as indicating a composition of about 95 per cent of some substance *a*, with boiling point 158° C., specific gravity 0.865,

and index of refraction 1.4714 and about 5 per cent of some other material, *b*, with values of all properties considerably higher (say, specific gravity 0.95, index 1.489). The gradual increase in the values of all the physical properties from the first fraction up to 95 per cent might be thought to be due to a gradually increasing proportion of this high-boiling and heavy material in the distillate (as was the case in the boiling-point curves of fig. 3); but this interpretation is shown to be incorrect by a procedure to be described—a procedure which was carried through for an entirely different purpose (see p. 25).

Figure 13 gives the result of a distillation of a part of the turpentine used in figure 2, which had been redistilled several times in order to remove the high-boiling and heavy material originally present. The curves show that this heavy material, *b*, has been entirely removed, and yet the values for all the physical properties increased to some extent as the distillation progressed, which indicated that the distillate was not a pure substance. Evidently, then, the gradual increase of these values in figure 2 was not due to the presence of *b* in the first 95 per cent of the distillate, but instead there must have been present another substance, *c*, with physical properties only slightly higher than those of *a*, the boiling points of *a* and *c* being so close together that the substances are very incompletely separated by distillation.

This interpretation agrees with the results of recent investigations of the chemical constituents usually present in turpentine. According to Semmler,¹ there may be present in American turpentine besides pinene² two other terpenes, camphene and nopinene³. The physical properties of these two terpenes have not been accurately determined, but it is sufficient to know that they are all higher than those of pinene. The first 95 per cent of the distillate from this turpentine was probably composed of a mixture of pinene with either camphene or nopinene, or both, and the last 5 per cent was made up of materials with much higher physical properties, probably oxidation products of the terpenes.

In the distillation of turpentine by this method the separation between the terpenes and the high-boiling nonterpenes is sufficiently complete, so that the physical properties of the first fractions are not appreciably influenced by the presence of materials with boiling points of from 30° to 35° higher. The mixture of the terpenes—pinene, camphene, and nopinene—is, however, not sufficiently well separated to make it possible to estimate the proportions of the dif-

¹ "Die Aetherische Oele," Vol. II, pp. 55 and 274.

² Boiling point, 155°–156° C.; specific gravity, 0.863; index of refraction, 1.4678.

³ The physical properties of both about the same at the following values: Boiling point, 160° to 165°; specific gravity, 0.874; index of refraction, 1.4724.

ferent constituents, and in this paper the three together will be designated as "pinene," unless for a special reason it is necessary to mention the other constituents.

DISTILLATION CURVES OF A WOOD TURPENTINE.

The curves obtained by the distillation of a wood turpentine (fig. 54) show it to be so different in composition from the gum turpentine just discussed that new points in the interpretation of the curves are introduced. The first 70 to 75 per cent of the distillate could well be, from the physical properties, a mixture of the same terpenes which are the main constituents of the turpentine shown in figure 2, the slightly lower physical properties being accounted for by smaller proportions of either nopinene or camphene, or both. But between the points where 70 and 95 per cent distill, the specific gravity of the distillate decreases, while the boiling point and index of refraction increase. This indicates the presence of some substance with higher boiling point and higher index of refraction, but with lower gravity than pinene. There is a terpene, dipentene, whose physical properties correspond well with those required by this substance; it has a higher boiling point (175° C.), a lower specific gravity (0.849 at 15° C.), and higher index of refraction (1.471 at 15° C.) than pinene; it is known to be one of the products of the action of high temperatures on pinene;¹ it has been identified as one of the constituents of wood turpentines produced in Finland and Russia;² and its presence in American "pine tar oil" has been indicated.³ It is quite probable, therefore, that the substance whose presence is indicated by the marked decrease in specific gravity of the fractions as the boiling point approaches 175° C. is dipentene and that it is formed from pinene by the high temperature employed in removing the turpentine from the wood.

CHEMICAL IDENTIFICATION OF DIPENTENE.

In order to make sure that dipentene was present in this turpentine, however, an identification by chemical means was carried out. By repeated fractional distillation and final distillation over sodium a considerable quantity of material was obtained with boiling points 172° to 174.1° C., specific gravity 0.8527 at 15° C., and index of refraction 1.4771 at 15° C. Part of this material was treated with bromine in glacial acetic acid⁴ and crystals were obtained which, after six crystallizations from ethyl acetate, melted at 125° to 125.5° C. (dipentene tetrabromide 125° to 126° C.), thus substantiating the con-

¹ Wallach, *Ann. Chem.* 227, 282.

² Aschan and Hjelt, *Chem. Ztg.* 18, 1566.

³ Kremers, *Pharm. Rev.* 22, 150.

⁴ Heusler-Pond, "Chemistry of the Terpenes," pp. 73 and 95.

clusions regarding the presence of dipentene, which were originally drawn from the physical properties. It seems very likely, therefore, that when a turpentine obtained from the long-leaf pine is distilled by the method described above, a decrease in specific gravity as the boiling temperature approaches 175° C. is a good indication of the presence of dipentene.

COMPLETENESS OF SEPARATION OF CONSTITUENTS.

The exact percentage of dipentene can not be determined from the curves in figure 54, since the separation between the pinene and the dipentene is not sufficiently sharp. There are small quantities of dipentene in the portion distilling between 70 per cent and 80 per cent as shown by the slight decrease in specific gravity, but it is only in the portion coming over after 80 per cent has been distilled that there are comparatively large quantities of dipentene present (as shown by the decided changes in direction in the boiling point and index of refraction curves). The separation between the dipentene and the "heavy oils" is, however, quite complete, and the percentage of the latter can be determined with considerable accuracy.

This turpentine contains, then, 95 per cent of terpenes (containing approximately 80 per cent pinene and 15 per cent dipentene) and 5 per cent of heavy, high boiling oils, probably nonterpenes.

COMPARISON OF FRACTIONATIONS BY DIFFERENT METHODS.

In order to show the advantages gained by the use of a dephlegmator, and by the use of direct distillation instead of steam distillation, the following distillations were carried out on the same turpentine: (1) Steam distillation without a dephlegmator; (2) direct distillation without a dephlegmator; (3) steam distillation with a

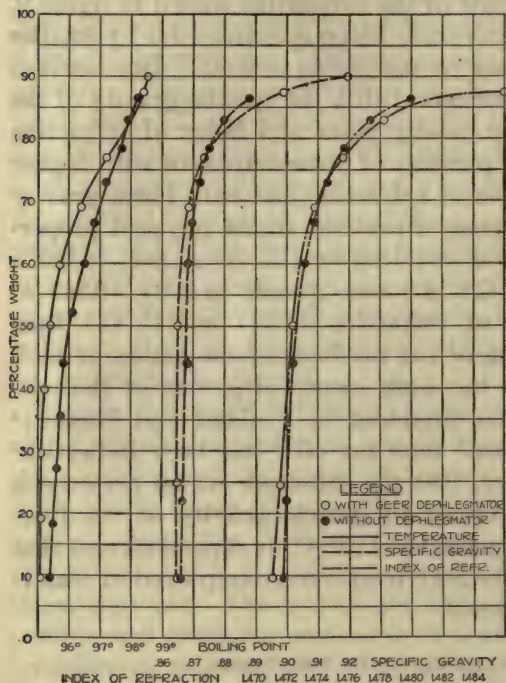


FIG. 4.—Comparison of fractionation obtained in steam distillation with a Geer dephlegmator and without a dephlegmator.

Geer dephlegmator as described in Forest Service Circular No. 152; and (4) direct distillation through a five-bulb LeBel-Henninger dephlegmator. The last-named dephlegmator will not be described, because another form was afterwards adopted as most suitable for general work.

STEAM DISTILLATION WITH AND WITHOUT A DEPHLEGMATOR.

In figure 4 are plotted together on one sheet for better comparison the curves obtained by steam distillation, both with and without a dephlegmator. The curves showing the results obtained by the use of a dephlegmator indicate the better fractionation, since they are more nearly vertical and have lower values over the first part of the distillation, and the breaks in the general direction are more marked than in the curves which give the results obtained by steam distillation without a dephlegmator.

DIRECT DISTILLATION WITH AND WITHOUT A DEPHLEGMATOR.

A still greater effect obtained by the use of a dephlegmator is indicated in figure 5, where are plotted together the curves from two direct distillations, one with and one without a dephlegmator. Here the differences in the position and direction of the curves and therefore in the fractionation are even more marked than in figure 4.

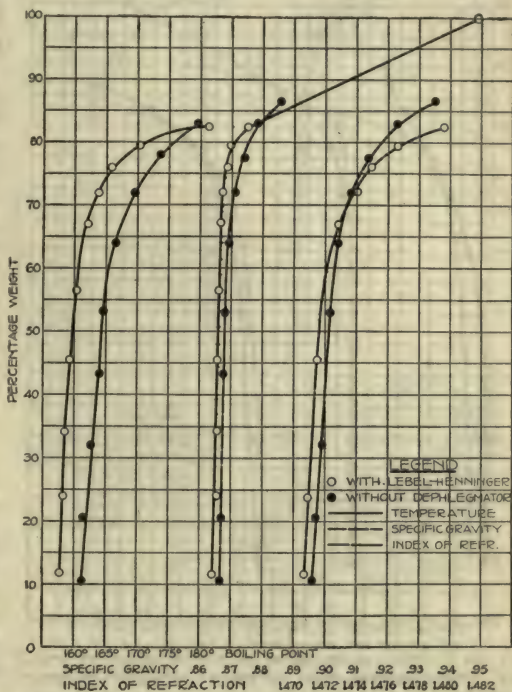


FIG. 5.—Comparison of fractionation obtained in direct distillation with a Le Bel-Henninger column and without a dephlegmator.

STEAM AND DIRECT DISTILLATION WITH DEPHLEGMATORS.

Figure 6 gives a comparison of the fractionation obtained by steam distillation through a Geer dephlegmator with that by direct distillation through a Le Bel-Henninger dephlegmator. In the direct-dis-

tillation curves there are all the evidences of better fractionation, i. e., lower values for the physical constants of the fractions over the first part of the distillation, more nearly vertical curves, and sharper breaks in the direction of the curves. On account of the better fractionation it is possible to judge the composition of the turpentine with a considerable degree of accuracy and it apparently contains very nearly 80 per cent of pinene, the remainder being heavy oils with all the physical properties much higher. From the steam-distillation curves, on the other hand, it would be impossible to make an accurate

judgment of the proportion of pinene present in the turpentine, because the separations are so incomplete and the curves change direction so gradually. This distillate is practically pure pinene up to about 70 per cent, but for some distance beyond that point it is an indefinite mixture of pinene with heavier and higher boiling materials.

Similar distillations were made on another kind of turpentine (refined turpentine No. 5) giving even more striking differences in the separations obtained. Figure 7 shows the results obtained by fractionation of three samples of the same turpentine (1) by steam distillation with a Geer

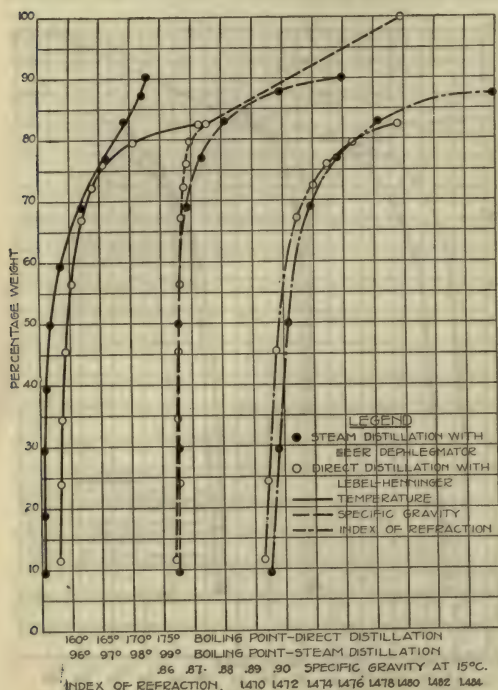


FIG. 3.—Comparison of fractionation obtained by steam distillation with a Geer dephlegmator and direct distillation with a Le Bel-Henninger dephlegmator.

dephlegmator, (2) by direct distillation without a dephlegmator, and (3) by direct distillation with a Le Bel-Henninger dephlegmator, the three sets of curves being plotted together on one sheet for easier comparison. The curves obtained by steam distillation with a dephlegmator, and by direct distillation without a dephlegmator show very similar characteristics; there are no rapid changes in direction until about 90 per cent is reached and then there is a rapid increase in all the physical constants; that is, these curves indicate about 90 per cent pinene and about 10 per cent of material with all physical constants higher. The curves obtained by direct distillation with a dephlegmator are, however, quite

different; they indicate that there are three main constituents of this turpentine sufficiently different in boiling points and other properties so that approximate determinations can be made of the proportion of each, these proportions being about 80 per cent of pinene, 15 per cent of dipentene, and 5 per cent of heavy residue. In the distillation by the first two methods the dipentene was not sufficiently well separated from the other constituents so that its presence was sharply indicated, the very slight and very gradual decrease in the specific gravity over the first 80 per cent of the distillation being the only sign of the presence of such a substance.

Another comparison of the results obtained by steam and direct distillation, both through a dephlegmator, is given in figure 8, in this case on a still more complex turpentine. The curves obtained by the direct distillation with a Le Bel-Henninger dephlegmator indicate that this turpentine is composed of about 11 per cent "light oils" (with boiling points below those of pinene), about 32 per cent of pinene, about 22 per cent of dipentene, and 35 per cent of heavy oils. The

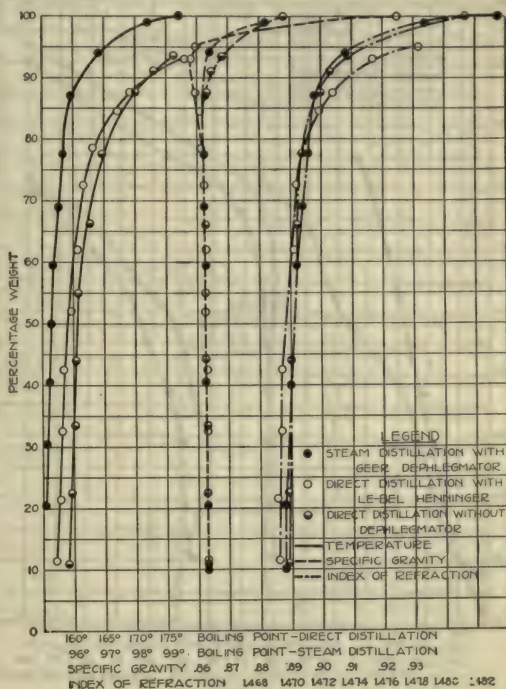


FIG. 7.—Comparison of fractionation obtained by (1) steam distillation with a Geer dephlegmator, (2) direct distillation with a Le Bel-Henninger dephlegmator, and (3) direct distillation without a dephlegmator.

separation between the pinene and the dipentene was not very sharply indicated—that is, the breaks in the direction of the curves are not very marked—but the amount of light oils, of turpentine oils (pinene and dipentene), and of heavy oils are quite accurately determined. In the steam-distillation curves, however, no sharp separation of the turpentine into its constituents are shown; that is, there are no sharp breaks in the curves. The presence of dipentene is not even indicated qualitatively, and the proportion of heavy oils (as in fig. 6) can not be judged within 8 to 12 per cent.

These examples of the results obtained in the fractionation of three different turpentines by different methods of distillation have shown

very clearly that, so far as the completeness of the separations and the corresponding ease and accuracy of the interpretation of the results are concerned, direct distillation through a dephlegmator is the best distillation method for the analysis of turpentine.

CHANGES IN COMPOSITION DURING DISTILLATION.

A possible objection to direct distillation with dephlegmators¹ is

that the higher temperatures required are likely to decompose some of the oil during the distillation so that the fractions will not represent the true composition of the original material. In those distillations in which the same turpentine was distilled direct and by steam there were no evidences of decomposition in any case except that the residues left in the distilling flask were usually darker in color when direct distillation had been used; the differ-

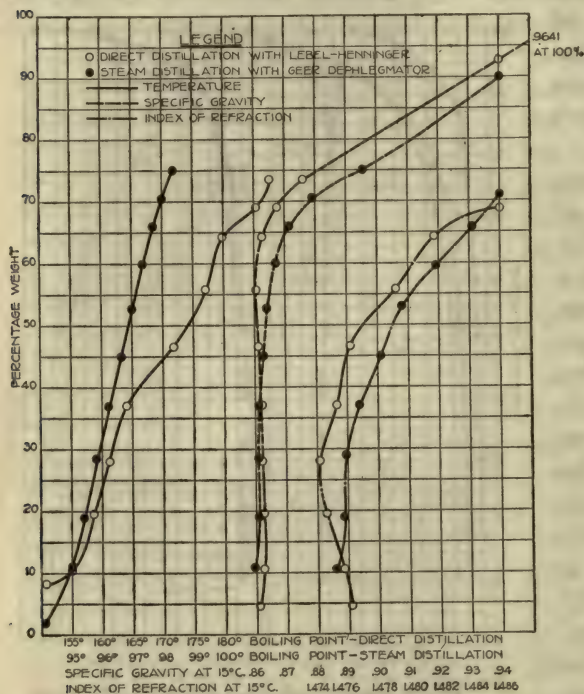


FIG. 8.—Comparison of fractionation obtained by direct distillation with a Le Bel-Henninger dephlegmator and steam distillation with a Geer dephlegmator.

ences in the curves obtained by the two methods were of such a nature as would indicate only a different fractionation.

CHANGES IN PHYSICAL PROPERTIES AT CONSTANT TEMPERATURES.

CRUDE WOOD TURPENTINE.

In order to obtain some idea of the comparative effects of different methods of distillation on the physical properties of turpentines, two samples of the same turpentine (turpentine No. 4) were boiled under conditions similar to those of steam and direct distillation—that is,

¹ This objection is mentioned in Forest Service Circular 152, "The Analysis of Turpentine by Fractional Distillation with Steam," by William C. Geer.

with and without water—and the specific gravity and index of refraction were determined at regular intervals; in both cases a well-cooled reflux condenser was used so that the loss was negligible. The effects of boiling under these two sets of conditions are shown graphically in figure 9. By boiling with water at a temperature of about 94°C ., the specific gravity of the turpentine was increased very slightly the first hour and after that decreased very slightly every hour until the boiling was stopped. By the boiling without the presence of water at a temperature of about 162°C ., the gravity increased at the rate

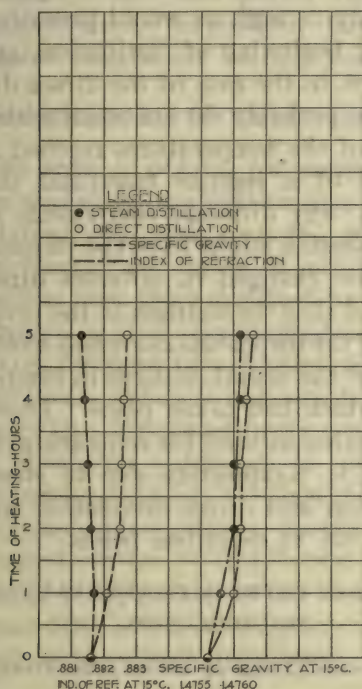


FIG. 9.—Effect of boiling with water (steam distillation) and of boiling without water (direct distillation) on the specific gravity and index of refraction of crude wood turpentine.

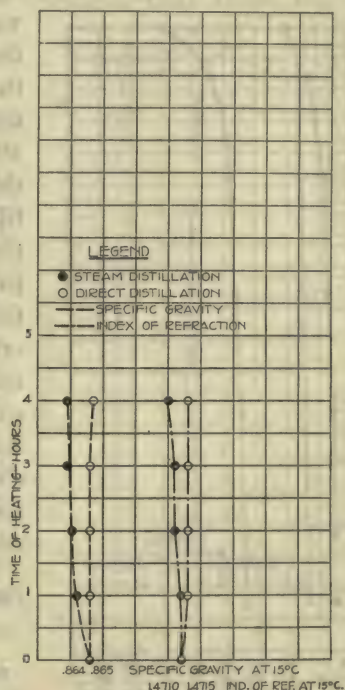


FIG. 10.—Effect of boiling with water (steam distillation) and of boiling without water (direct distillation) on the specific gravity and index of refraction of refined wood turpentine.

of 0.00045 per hour for the first two hours, and then at a much slower rate. The index of refraction was increased considerably by both methods of boiling during the first two hours and then more slowly.

REFINED WOOD TURPENTINE.

The results from a similar series of experiments on another turpentine (turpentine No. 5, the analysis of which is shown in fig. 7) are expressed graphically in figure 10. Here the changes in the physical properties due to methods of boiling are less than were shown in

figure 9, being within the limit of possible experimental errors. But the same general tendency of the specific gravity to be slightly increased by the direct boiling and slightly decreased by the boiling with water is shown, as in figure 9. The index of refraction is, however, slightly decreased by the boiling with water instead of being increased.

The effect of the boiling on the physical properties of the turpentines is apparently not very great in either case, but for two reasons the results obtained are not conclusive: (1) The temperatures to

which the turpentines were subjected were only as high as would prevail at the very beginning of distillation, and therefore, in the case of the direct distillation, probably do not come within 40° C. of the temperatures reached at the end of a regular fractional distillation; (2) different components of the turpentine might have the physical properties changed in different directions and thus the changes in the properties of the turpentine as a whole would not show the actual changes in composition which had taken place. Therefore, another method for comparing the changes in composition due to steam distillation and direct distillation was carried out as described below.

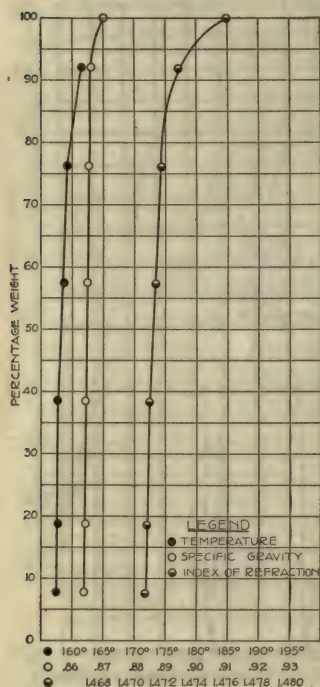


FIG. 11.—Alteration in physical properties of a crude wood turpentine due to direct distillation.

CHANGES IN PHYSICAL PROPERTIES UNDER FRACTIONATION.

DIRECT DISTILLATION OF CRUDE TURPENTINE.

Crude turpentine No. 4 was repeatedly redistilled by direct distillation through a Le Bel-Henninger dephlegmator. Each time the heavy residue left in the flask and as much of the distillate as showed especially high physical properties were rejected. The first distillation was similar to the direct distillation shown in figure 6, and the fractions up to 79.5 per cent formed the material for the next distillation, the residue and the last fraction of the distillate being rejected. The curves showing the course of the third and fourth distillations were practically identical and are both represented by the curves in figure 11. All the heavy oils present in the original turpentine must have been removed by the first two distillations.

otherwise the physical properties of the residue would have been higher in the third distillation than in the fourth. Since the original heavy oils had been completely removed, the higher physical properties of the residue in the third and fourth distillations must be due to the heavy oils formed during the course of the distillations. The difference in the values of the physical constants between the last fraction and the residue in figure 11 is therefore an approximate measure of the change in composition due to the temperature of the distillation. The lack of any marked difference in properties between the first and second fractions indicates that there was no appreciable decomposition into substances with lower boiling points.

STEAM DISTILLATION OF CRUDE TURPENTINE.

In order to obtain, for comparison, the change in composition due to distillation with steam, all the material represented by figure 2, except the residue, was twice distilled by steam through a Geer dephlegmator. The curves for these two distillations were practically identical and are shown in figure 12. Reasoning in the same way as above the difference in physical properties between the last fraction and the residue indicates the change in composition due to distillation. This change is appreciable in the case of the steam distillation, but is somewhat less than in the direct distillation.

DIRECT DISTILLATION OF GUM TURPENTINE.

The same distillation was made on a gum turpentine (the one whose analysis is shown in figure 2). In this, as in case of the wood turpentine, the third and fourth distillations gave nearly identical curves, which are shown in figure 13. The amount of change in composition is also very nearly the same as in the case of the wood turpentine shown in figure 11.

It is evident, then, that, under the conditions prevailing in the distillation shown in figures 11 to 13, the amount of change in composition during analysis is very slight, either in steam or direct distillation. This change apparently consists in the polymerization of a portion of the terpenes to substances with higher physical constants,

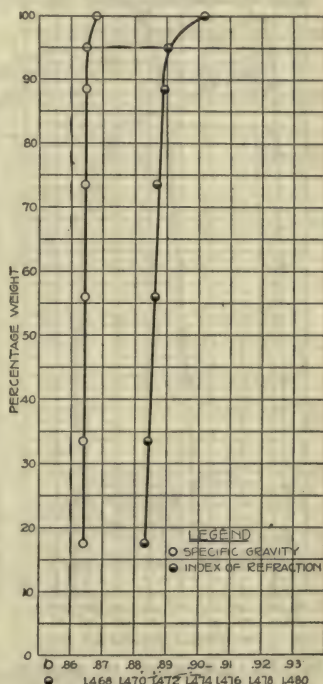


FIG. 12.—Alteration in physical properties of a crude wood turpentine due to steam distillation.

and seems to take place mostly during the very last part of the distillation when the amount of oil in the distilling flask has become very small and when there is, therefore, a better chance for the sides of the flask to become superheated.

CHANGES IN COMPOSITION AND INTERPRETATIONS OF CURVES.

TURPENTINES CONTAINING NO HEAVY OILS.

When a turpentine containing no heavy oils is distilled by direct

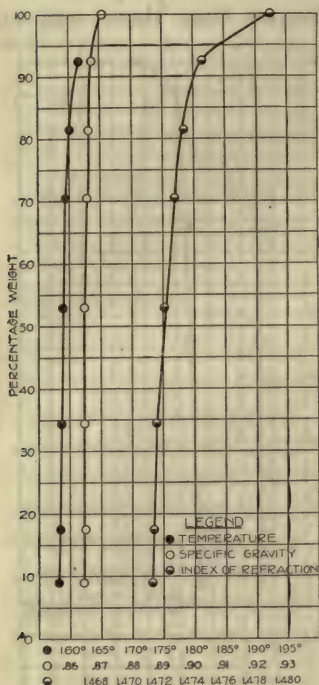


FIG. 13.—Alteration in physical properties of a gum turpentine due to direct distillation.

heat the undistilled residue left in the flask will contain a small amount of heavy oils, owing to changes occurring during the distillation, and the specific gravity and index of refraction of this residue will be higher than those of the last distilled fraction; the curves will, therefore, seemingly indicate that small amounts of heavy oils were present in the original turpentine, but from the curves of figures 11 to 13 the effect of the changes, which occurred during the distillation, can be determined, and the interpretation of the curves can be altered to correspond. For instance, in the distillation of 95 per cent of a turpentine containing no heavy oils, the production of heavy oils during the distillation will be sufficient to increase the gravity and index of the residue of 5 per cent by 0.0035 and 0.0040, respectively. Of course, if the residue is made smaller—say 2 per cent—then the apparent effect would be greater, since there is a chance for more polymeriza-

tion during the distillation of that portion between 95 and 97 per cent; and the proportion of heavy oil to turpentine oil in the undistilled residue is increased—and hence the physical properties increased—as the distillation progresses. It is therefore necessary to leave about 5 per cent of oil undistilled in order to be able to correct accurately for the effect of polymerization.

TURPENTINES CONTAINING HEAVY OILS.

When, however, the turpentine to be distilled contains some heavy residue (as for instance in fig. 2) the conditions are different; the terpenes may all be distilled over, and the residue left in the flask

would then be all heavy oils. These heavy oils might be changed by the heat, either more or less than the terpenes were in figures 11 to 13, but there is really no necessity for determining this change accurately because a small change in the physical properties does not effect the interpretation of the curves. For instance, if in figure 2 the gravity of the residue had been 0.930 instead of 0.945, the composition of the turpentine as interpreted from the curves would still have been 95 per cent pinene and 5 per cent heavy residue; that is, a gravity of 0.93 is so much higher than that of the terpenes that the presence of a considerable amount of terpenes in the residue is excluded; and the only possible interpretation of the composition of the residue is that it does not contain terpenes in considerable quantities.

The change in composition of a turpentine during analysis by direct distillation is, therefore, without effect on the interpretation of the results, except in case there is no high boiling and heavy residue present in the original material; and in this case the approximate effect is known and the results can be corrected.

CHOICE OF A DEPHLEGMATOR.

The Le Bel-Henninger dephlegmator has been used with good results in many turpentine analyses; it is efficient in fractionating power, and when properly constructed gives no trouble by filling up with liquid; the only objections to it are that it is expensive and fragile. The simplest, least expensive, and least fragile dephlegmator is of the Hempel column type, and several different sizes of Hempel columns were tried to find if a practicable sized Hempel would give as good separations as the Le Bel-Henninger. It was found that the Hempel column, shown in figure 1, gave slightly better separations than the five-bulb Le Bel-Henninger and a com-

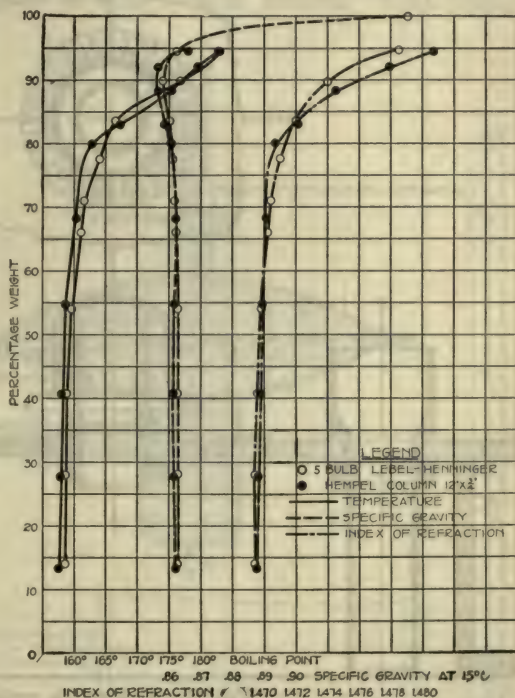


FIG. 14.—Comparison of fractionation with Le Bel-Henninger and Hempel column dephlegmators.

parison between the results obtained by the use of these two dephlegmators is shown in figure 14. The main difference between the curves obtained by the two types of dephlegmators is in the specific gravity in the vicinity of 90 per cent; the fractions obtained by distillation through the Hempel column have a lower gravity over this part of the curve, indicating a purer dipentene and therefore better fractionation; the sharper break in the curves at about 80 per cent also indicates better fractionation. On account of the simplicity and cheapness of the Hempel column as well as its better fractionation, it is recommended as the preferable dephlegmator for turpentine

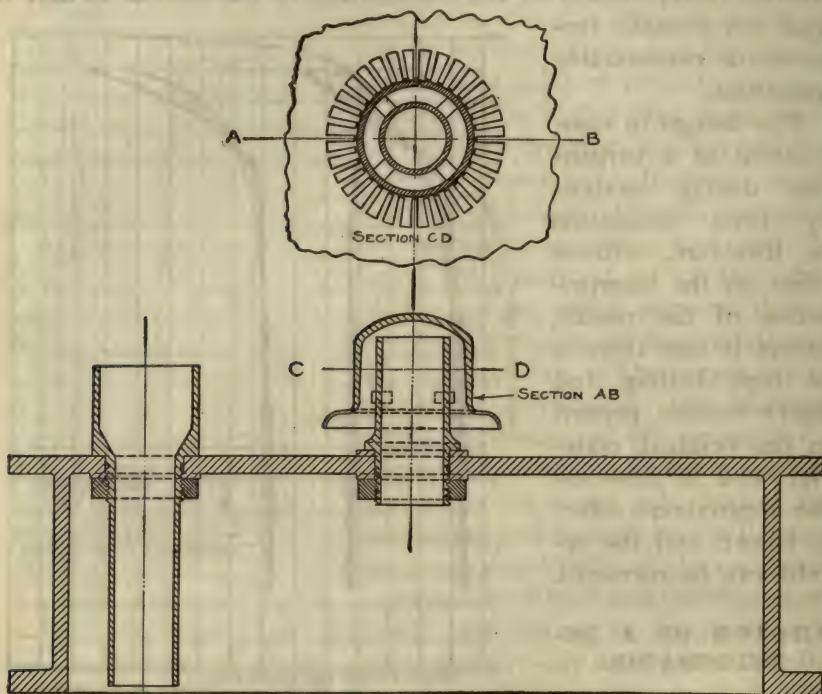


FIG. 15.—Boiling cap section.

distillations. This type of dephlegmator has been used in making many of the distillations recorded in the following pages, and has given very satisfactory results.

REFINING.

Five samples of crude wood turpentine were refined; these were collected from commercial plants using widely varying processes, a brief description of which will be given. The apparatus used in the refining and the method of conducting the distillations were similar for all the samples.

DESCRIPTION OF APPARATUS.

The refining still was of the continuous type with two phases, giving two distilled products and one undistilled residue. It was designed by the makers especially for this work, since stills of this type for the refining of turpentine had not previously been used or manufactured in this country.

BOILING-CAP SECTIONS.

The boiling-cap section, shown diagrammatically in figure 15, is the typical and important division of the column, the other kinds of sections being modifications of this typical section. Each section

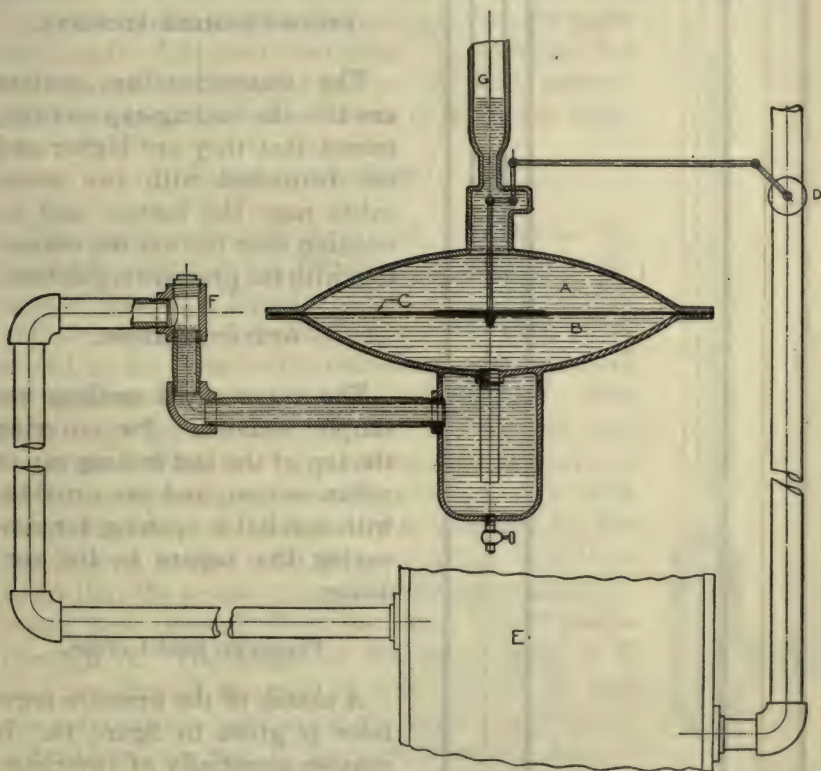


FIG. 16.—Pressure regulator.

is 14 by 12 inches inside diameter, and is supplied with eight boiling caps and two return pipes. The liquid level on each section is kept even with the top of the return pipes, and the vapors from the section below are made to pass through this liquid by means of the boiling caps; the rim of each cap is slotted so that the vapor passes through the liquid in small bubbles, and good contact between the ascending vapor and the descending liquid is thus obtained.

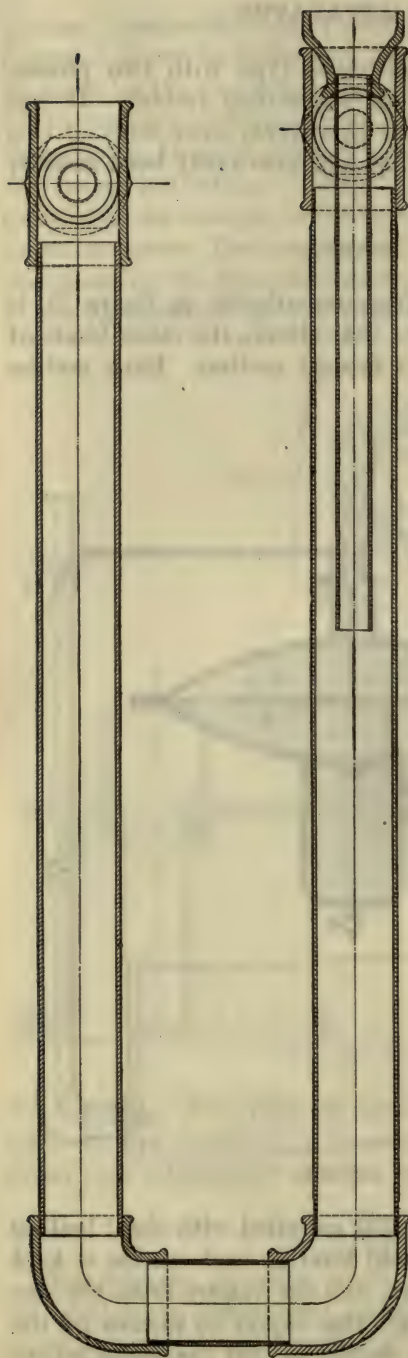


FIG. 17.—Separator.

REFLUX-CONDENSER SECTIONS.

The reflux-condenser sections are really a combination of a boiling-cap section with a reflux condenser; that is, they are similar to figure 15, except that below the return pipes there is a chamber filled with copper tubes, around which the vapors must pass and through which water is kept flowing.

STEAM-CHAMBER SECTIONS.

The steam-chamber sections are like the boiling-cap sections, except that they are higher and are furnished with two steam inlets near the bottom and an opening near the top for connection with the pressure regulators.

OUTLET SECTIONS.

The vapor-outlet sections are simply chambers for covering the top of the last boiling cap or reflux section, and are provided with a suitable opening for conveying the vapors to the condenser.

PRESSURE REGULATORS.

A sketch of the pressure regulator is given in figure 16. It consists essentially of two chambers, A and B, separated by a rubber diaphragm. Any motion of the diaphragm such as might be caused by a change of pressure in either chamber is communicated by the system of levers to the steam valve D. For in-

stance, if there is an increase of pressure in the steam chamber E, there is a corresponding increase of pressure on the bottom of the diaphragm and the diaphragm moves upward, closing the valve D until the pressure is again normal in E.

SEPARATORS.

The separator, figure 17, is the same in principle as the ordinary automatic oil and water separator used in practice, but it is made much more compact in order that only a small amount of oil can be retained in it at one time. This apparatus can be made entirely from ordinary iron pipe and pipe fittings. A separator like that shown in the drawing, with a total length of 3 feet, and with the main tube made of $2\frac{1}{2}$ -inch iron pipe, was sufficient for the separation of 20 to 23 gallons of refined turpentine from about the same amount of water per hour.

SPEED INDICATORS.

The speed indicator,¹ figure 18, is based on the principle that there is a definite relation between the rate of flow of a liquid through a small aperture and the pressure of the liquid at the aperture. The liquid, as for instance the refined turpentine, enters the indicator from the separator through A. The only outlet for the liquid is the tube C, which may be entered from the top or through the aperture D, this aperture is made small enough so that, with the normal flow of the liquid, it is necessary for the liquid level to rise some distance in the tube B in order that the pressure may be sufficient to make the liquid flow through D at the same rate it enters through A. The height of the liquid level in B is a measure of the rate of flow through D, and the relationship between these factors is obtained by calibration; that is, by measuring the rate of flow from C while the liquid level is maintained at constant measured distances above D. The calibration curve for the crude-turpentine indicator is shown in figure 19.

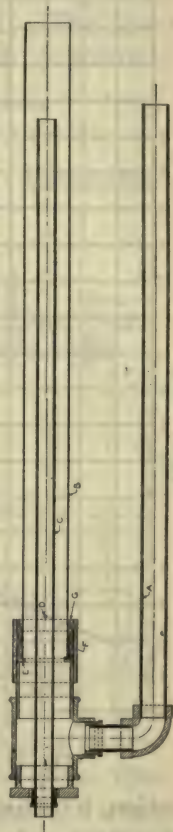


FIG. 18.—Speed indicator.

¹ This apparatus can be made almost entirely from ordinary iron pipe and pipe fittings. The pipe C should, however, be of brass so that the aperture D may not be changed in size by rusting and, of course, the tube B must be of glass. The method of fastening the glass tube into the iron parts is shown in the drawing; E is a support of tin plate or other thin, easily shaped, metal soldered to the nipple in order to support the tube B and packing F; G is a thin layer of litharge and glycerin cement, which makes the joint oil or water-tight.

The other parts of the apparatus were of standard design and require no special explanation.

GENERAL ARRANGEMENT AND OPERATION OF APPARATUS.

A diagram of the general arrangement of the apparatus is given in figure 20. The fractionating column A is divided into the two separate columns A_1 and A_2 at the place marked by the heavy line. A_1 and A_2 are made up of the different kinds of sections already described, as follows: A_1 from the bottom upward, 1 steam chamber

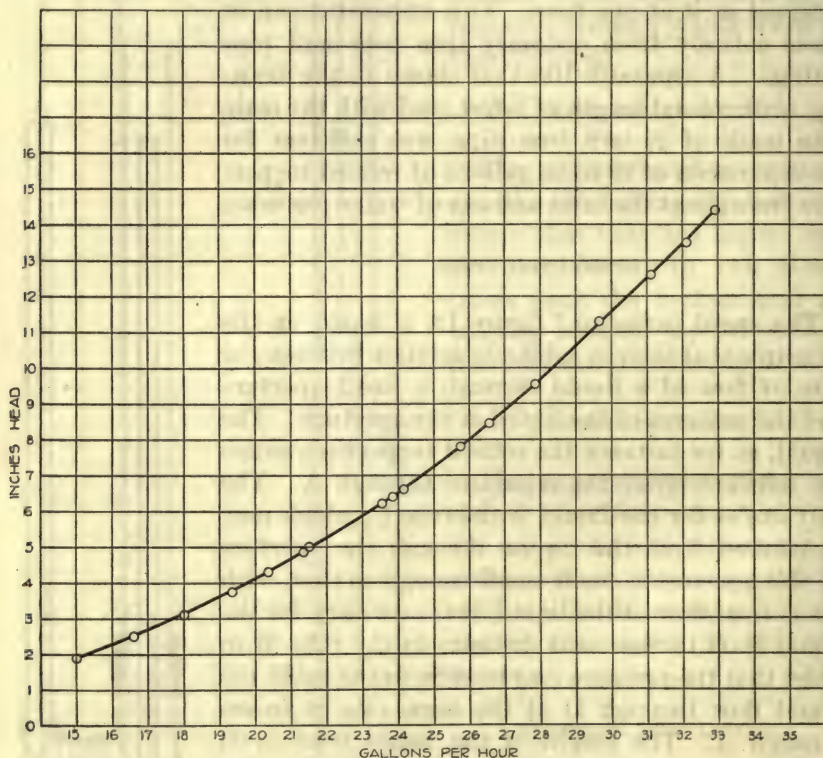


FIG. 19.—Calibration curve for crude turpentine speed indicator.

section, 5 boiling-cap sections, 2 reflux-condenser sections, and 1 outlet section. A_2 is similarly made up of 1 steam chamber section, 13 boiling-cap sections, 3 reflux-condenser sections, and 1 outlet section.

The tank for crude oil F is connected through the pump G with the reservoir D; the overflow pipe from D, through valve 9, furnishes an air outlet for D while it is being filled and serves the purpose also of notifying when D is full; the return pipe through valve 10 also makes it possible to draw out any material from D directly into the tank F. The reservoir D is simply a 110-gallon steel drum, arranged on the principle of a Mariotte's bottle in order to keep the flow of

crude turpentine through valve 1 constant, whatever the level of crude turpentine in D. The reservoir D having been filled and valve 10 closed, valve 1 is opened until the crude turpentine is running at the required speed as registered on the speed indicator J, say 25 gallons per hour. The crude turpentine then flows through the feed heater C, where it is heated by steam from valve 2 to a temperature of about 95°C ., and enters the upper column A_1 at the fourth boiling cap section as indicated. The steam supply is now adjusted by the pressure regulator H_1 , and the water in the reflux sections by valve 4, so that the distillate leaving the top of column A_1 is running at the required speed as indicated on the speed indicator J_2 . The speed required is determined from the analysis of the crude turpentines;

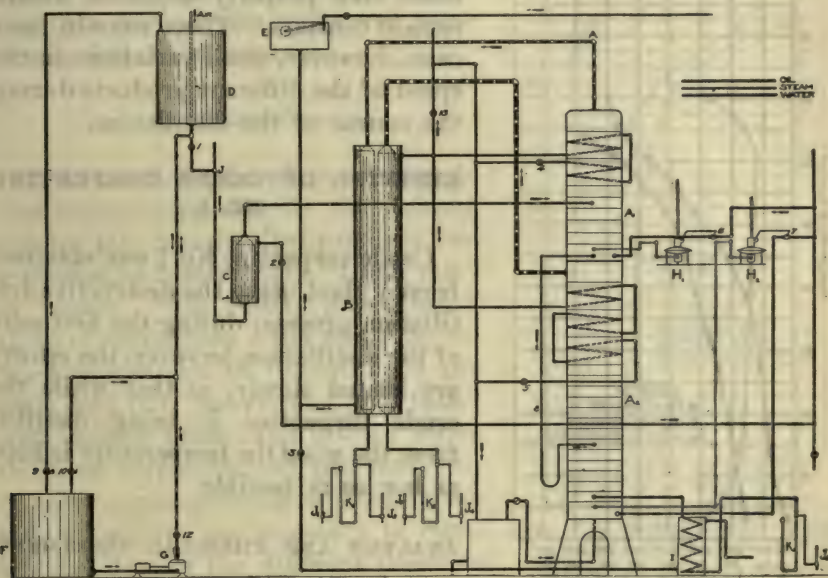


FIG. 20.—General arrangement of apparatus for refining crude turpentine.

for instance, if 5 per cent of low-boiling material which it is desired to remove from the refined turpentine is present in the crude, then it is only necessary to adjust the speed of the first distillate to 5 per cent of the speed of the crude.

The portion of the crude not distilled in column A_1 flows from the bottom section through the trapped pipe 8 into the column A_2 at the sixth boiling cap section. Here the distillation process is repeated as in the top column, the water and steam supplies being adjusted by valve 5 and the regulator H_2 , respectively, so that the distillate leaves the top of A_2 at the required rate.

The undistilled residue or "tails" runs out in liquid form from the first boiling cap section, is cooled in the tails cooler I, separated from

the water, and the speed measured in the indicator J_3 . The speed of the tails is not directly controlled, but is dependent upon the speeds of the other products, being equal to the speed of the crude, minus the sum of the speeds of the two distillates.

A constant head for the water supply is maintained by means of the float valve in the reservoir E, so that when the valves 4, 5, and 13 are once adjusted the flow of water through the condenser B and therefore through the refluxes of A_1 and A_2 will be constant. The pressure regulators H_1 and H_2 are used to maintain a constant pressure of steam in the bottom section of the columns A_1 and A_2 , respectively, and therefore the speed of the distillates, once properly adjusted, should remain constant. There were in most cases, however, small variations in the speed of the different products during the course of the distillation.

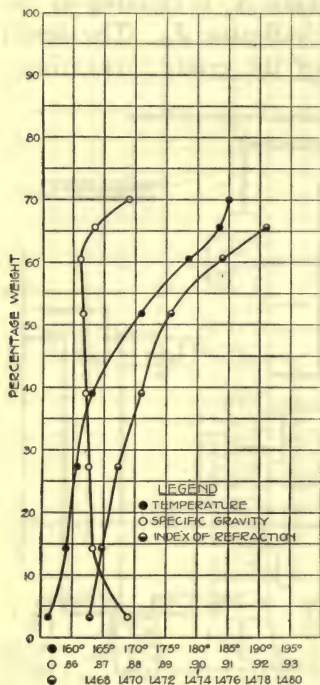


FIG. 21.—Boiling point, specific gravity, and index of refraction curves for crude turpentine No. 1, untreated.

these two things, in connection with the high gravity indicated the impossibility of obtaining by distillation alone any refined turpentine of good quality from this part of the crude, although the boiling points were mostly within the range of a good turpentine. Because it was probable that the contaminating material could be removed by treatment with alkali, the original crude turpentine was agitated with a warm caustic soda solution until action ceased.

The analysis of the treated crude is shown in figure 22. The gravity of the first few fractions was decreased and the index of refraction was increased by the treatment; the color and odor of all the fractions

REFINING OF CRUDE TURPENTINE NO. 1.

Crude turpentine No. 1 was obtained from a plant using the destructive distillation process; during the first part of the distillation, however, the retorts are heated slowly, so that while the crude turpentine is being distilled from the wood the temperature is kept as low as is feasible.

ANALYSIS AND CHEMICAL TREATMENT.

The analysis of the crude turpentine is given in figure 21. The odor of the first four fractions was very disagreeable, and the color was a dark yellow;

were also improved. The first fraction of the treated crude, however, had a more disagreeable odor and more color than the other fractions; the second fraction still retained these undesirable qualities, but to a lesser degree; the rest of the fractions were practically colorless and had only slight traces of the strong odor of the first fraction. Since that portion of the distillate which had the undesirable properties also had lower boiling points than the rest of the material, it seemed likely that the undesirable portion could be separated by distillation, with a consequent improvement in the properties of the refined turpentine.

The heavy oils began to come over in quantity at about 70 per cent and the distillate beyond this point was practically free from terpenes.

DISTILLATION.

It was decided therefore to separate this crude turpentine by the refining process into two distilled products and a residue, as follows: (1) "Light oils" or "heads" with boiling points below those of turpentine, 5 per cent; (2) refined turpentines or "hearts," 65 per cent; and (3) "heavy oils" or "tails" with boiling points and gravity above those of turpentine, 30 per cent. In order to secure such a separation in the refining still it was necessary to regulate the flow of the different products to the following speeds (the crude turpentine running at 25 gallons per hour):

For heads 5 per cent, of 25, or 1.25 gallons per hour.

For hearts 65 per cent of 25, or 16.25 gallons per hour.

For tails 30 per cent of 25, or 7.5 gallons per hour.

In the distillation of this turpentine and all others as well the first runnings of the distillates were caught separately until the adjustment was completed, because the composition of the products would vary greatly at first. The analyses shown hereafter represent the composition of the products obtained after the still was properly adjusted and running smoothly. For instance, this run on turpentine No. 1 occupied about $4\frac{1}{2}$ hours, but the products shown in the analyses were obtained during a little less than 4 hours of this time.

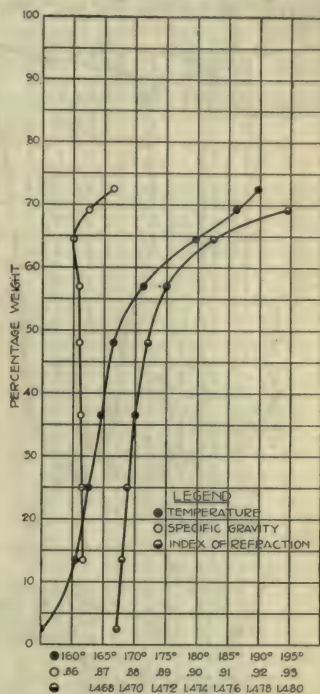


FIG. 22.—Boiling point, specific gravity, and index of refraction curves for crude turpentine No. 2, after treatment with caustic soda solution.

PRODUCTS OF DISTILLATION.

The analyses of the products obtained from the distillation of crude turpentine No. 1 are shown in figures 23, 24, and 25.

HEADS.

The heads (fig. 23) vary greatly in composition, and approximately the first 70 per cent have higher indices of refraction, and

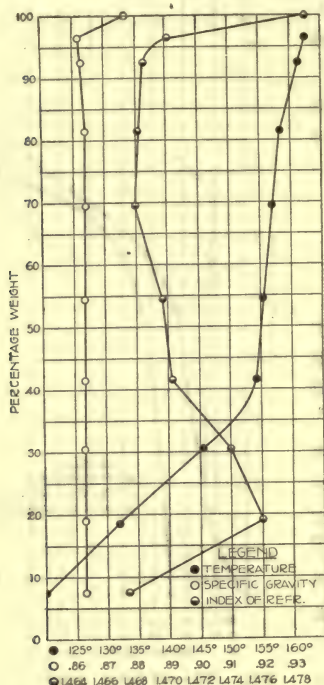


FIG. 23.—Boiling point, specific gravity, and index of refraction curves for "heads" obtained in refining of crude turpentine No. 1.

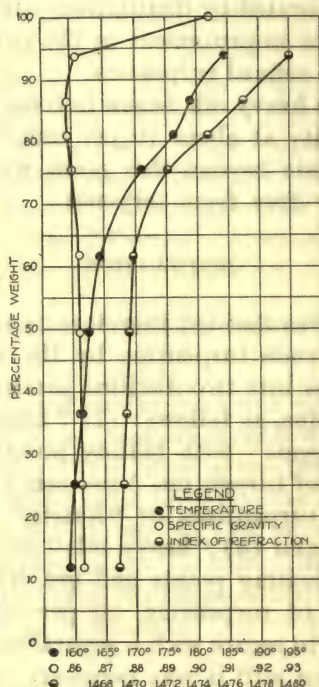


FIG. 24.—Boiling point, specific gravity, and index of refraction curves for "hearts" obtained in refining of crude turpentine No. 1.

lower boiling points, than the first fraction of the hearts; this portion of the distillate also had a darker color and more disagreeable odor than the fractions above 70 per cent. That portion above 70 per cent could well be included in the hearts without affecting the properties of the latter, but probably it is not possible to separate completely so small an amount of heads as were present in the crude, 2.5 to 3.0 per cent, without removing some of the hearts at the same time.¹

¹The heads, on account of the method of their production, could not contain any heavy oils, and the higher physical properties of the residue in this analysis (and in the analyses of all the other heads) must be due to polymerization during the distillation (see p. 26).

HEARTS.

The analysis of the hearts is shown in figure 24; there is apparently no material in the hearts with boiling points below 158°C ., and the color and odor of the first fractions were not markedly different from those of the next few fractions; that is, the separation of the heads from the hearts was complete. This separation of the low boiling constituents improved both the color and odor of the refined turpentine, making it almost colorless and leaving only a faint trace of the "destructive distillation" odor. There was a small amount, about 6 per cent, of heavy material in the hearts, showing an incomplete separation from the tails.

TAILS.

The tails were analyzed (fig. 25) only to determine how good a separation was obtained between the tails and the hearts. All the properties of the first fraction (9 per cent) of the tails were higher than any fraction of the hearts, except, of course, the residue. But since the composition of the distillate was changing rapidly at this point (the curves all slanting decidedly), it is probable that a small amount of this first fraction was composed of material with properties the same as some portion of the hearts; perhaps 3 to 4 per cent of the tails was composed of materials which should have been in the hearts.

REFINING OF CRUDE TURPENTINE NO. 2.

Crude turpentine No. 2 was produced at a plant using "lightwood" as a raw material. The process of distillation was the steam process and was different from the usual steam process only in the size of the chips, which were smaller, and in the greater steam pressure used, which was about 50 pounds.

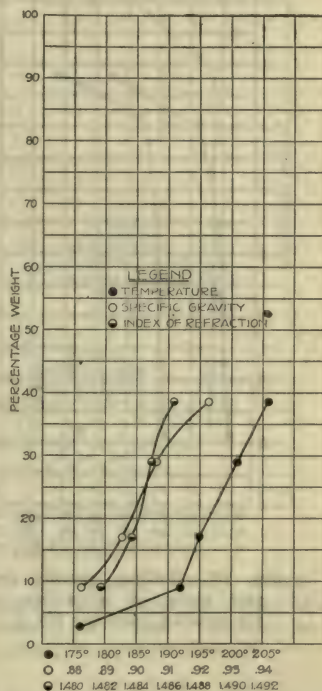


FIG. 25.—Boiling point, specific gravity, and index of refraction curves for "tails" obtained in refining of crude turpentine No. 1.

ANALYSIS.

The analysis of this crude is shown in figure 26. There were no evidences in this crude of low boiling material similar to that in

crude turpentine No. 1, but the odor of the first fraction was quite different from the second, and it was decided to separate a small amount of heads in order to find if the odor of the hearts was improved. This crude turpentine is seen to contain about 60 per cent pinene, 8 per cent dipentene, and 32 per cent heavy oils. It was decided to attempt to obtain about 4 per cent of heads, which would leave about 64 per cent of turpentine material to be separated as hearts.

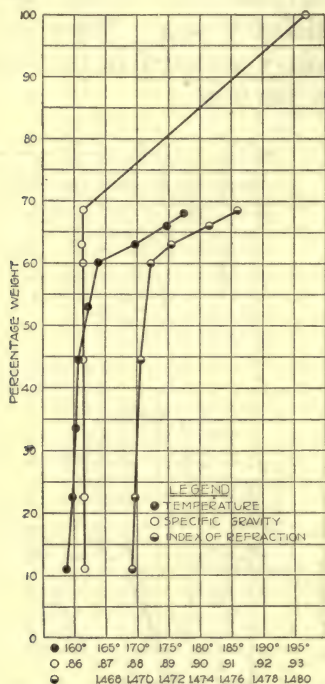


FIG. 26.—Boiling, specific gravity, and index of refraction curves for crude turpentine No. 2.

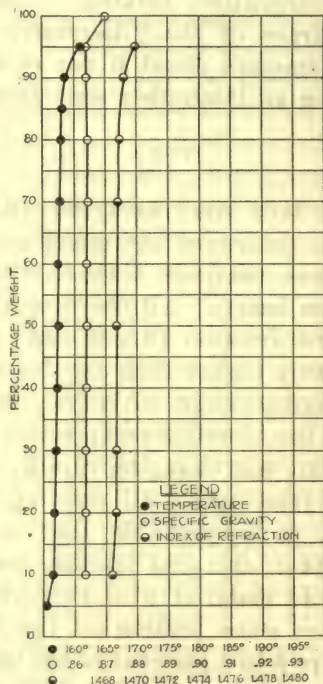


FIG. 27.—Boiling point, specific gravity, and index of refraction curves for "heads" obtained in refining of crude turpentine No. 2.

DISTILLATION.

The crude was run into the still at a speed varying from 24.8 to 25 gallons per hour; the heads were kept as close as possible to 1 gallon per hour (varying from 0.5 to 1.5 gallons per hour), and the hearts varied from 15.7 to 16.1 gallons per hour. Except for the rather large variation in the heads, the distillation seemed to run smoothly.

PRODUCTS.

The analyses of the products are given in figures 27, 28, and 29.

HEADS.

Although the difference in odor between the heads and the hearts was quite noticeable, they did not show any great difference in other properties; the odor of the first 10 per cent of the heads was also somewhat different from the rest of the fractions. The amount of material present in these heads with properties different from the main part of the turpentine (hearts) is therefore very small in pro-

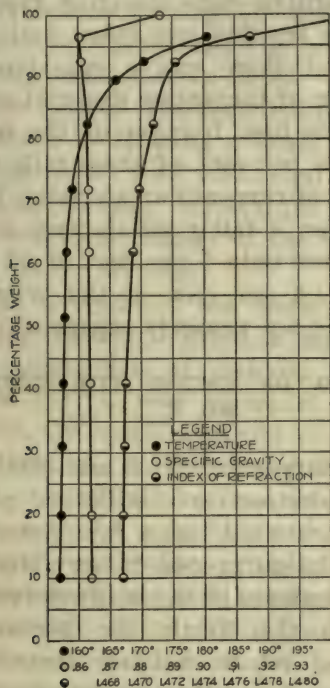


FIG. 28.—Boiling point, specific gravity, and index of refraction curves for "heads" obtained in refining of crude turpentine No. 2.

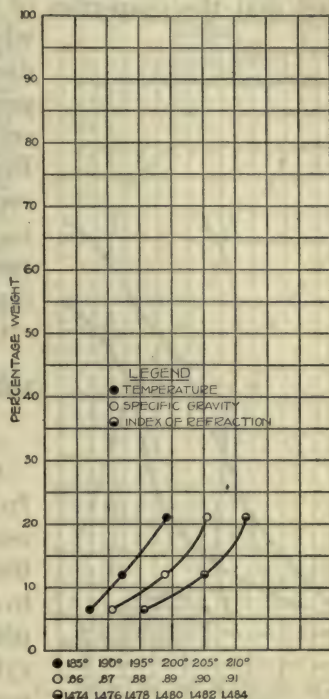


FIG. 29.—Boiling point, specific gravity, and index of refraction curves for "tails" obtained in refining crude turpentine No. 2.

portion to the original crude, so small that it is impracticable and really unnecessary to separate it.

HEARTS.

The hearts contain only about 3.5 per cent of heavy material, and the gravity of this portion is only slightly greater than that of the main part of the hearts, the gravities being 0.885 and 0.863, respectively, which shows a very satisfactory separation of hearts and tails.

TAILS.

The tails, however, contain a small amount of material which might have been included in the hearts. The properties of the first fraction of the tails (7 per cent) are higher than any portion of the hearts except the residue, but the difficulty of completely separating in one distillation a small amount of turpentine material, from a large proportion of higher boiling material like the "pine oil," and the fact that the properties of the distillate were changing rapidly

when this fraction was taken both indicate that there was a considerable proportion of turpentine material present in this first fraction of the tails. Perhaps 5 per cent of these tails was composed of turpentine materials. This is, however, a fairly satisfactory separation, since only 5 per cent of 32 per cent, or 1.6 per cent of the original crude, was not properly separated.

REFINING OF CRUDE TURPENTINE NO. 3.

Crude turpentine No. 3 was obtained from a destructive distillation plant using "lightwood" as a raw material; the distillation process is very similar to that employed by the hardwood retort plants in the North, the horizontal cylindrical retorts holding about one cord of wood and the complete distillation taking only 24 hours. The total distillate is condensed and collected together and the pyroligneous acid removed from the crude oil by settling.

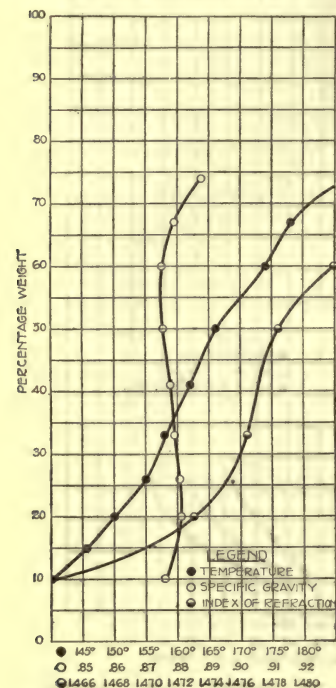


FIG. 30.—Boiling point, specific gravity, and index of refraction curves for crude turpentine No. 3, untreated.

The crude oil thus obtained is distilled with steam until nearly all the volatile oil is removed. The crude turpentine used in the refining experiments was a sample of the distillate obtained from this crude oil.

ANALYSIS AND CHEMICAL TREATMENT.

The analysis of the crude is shown in figure 30. It can be seen from the curves that this crude is a very complex mixture, since none of the properties remain at all constant over any considerable

portion of the distillation; a large part boils below the usual boiling point of turpentine, and the specific gravity over the complete range of the distillation is higher than any portion (except the residue) of a refined turpentine should be. All the fractions were also colored and had very disagreeable odors. It was seen that no separation by distillation alone could give a distillate suitable for a refined turpentine, and therefore it was necessary to give a chemical treatment before refining by distillation. The crude was therefore agitated with caustic soda solution at 55° C. until action ceased. The cessation of action is shown by treating a small amount of the crude with additional caustic soda at the same temperature without any resultant change in the color of the caustic solution.

The analysis of the crude treated in this manner is given in figure 31. The proportion of material boiling below 155° C. was decreased considerably, the specific gravity of all the fractions was decreased, and the color and odor of all fractions improved. The index of refraction curve was, however, somewhat irregular, and all the fractions still retained some disagreeable odor. That portion of the distillate between 11 per cent and 64 per cent was more suitable for a refined turpentine than that above or below, and it was therefore decided to separate the treated crude into 11 per cent heads, 53 per cent hearts, and 36 per cent tails.

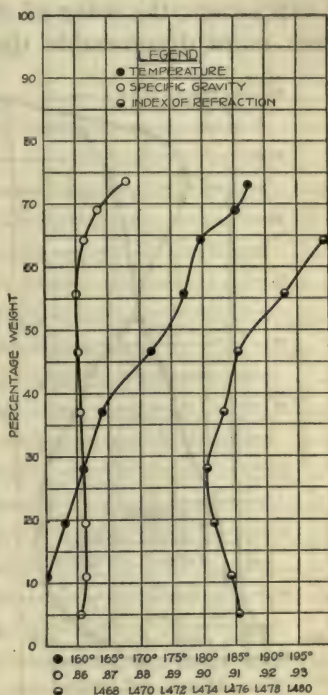


FIG. 31.—Boiling point, specific gravity, and index of refraction curves for crude turpentine No. 3, after treatment with caustic soda solution.

DISTILLATION.

The crude was run into the still at a speed of 25 gallons per hour, and the speeds of the heads and hearts were maintained respectively, as nearly as possible, at 11 per cent of 25, or 2.75 gallons per hour, and 53 per cent of 25, or 10.75 gallons per hour. During this distillation great difficulty was experienced in regulating the speed of the products, and the effects of this are seen in the composition of the products.

PRODUCTS.

The analyses of the products are shown in figures 32, 33, and 34.

HEADS.

The heads (fig. 32) contained about 80 per cent of material of complex composition, with widely varying boiling points and indices of refraction; the color of this 80 per cent was darker and the odor much more disagreeable than those of the last 20 per cent. The

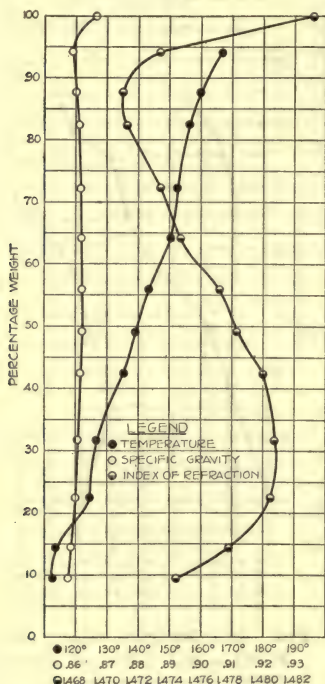


FIG. 32.—Boiling point, specific gravity, and index of refraction curves for "heads" obtained in refining of crude turpentine No. 3.

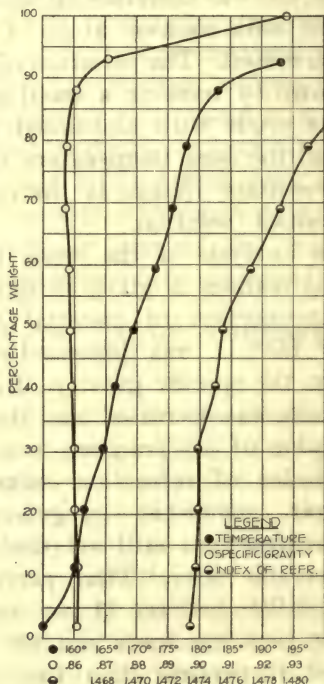


FIG. 33.—Boiling point, specific gravity, and index of refraction curves for "hearts" obtained in refining of crude turpentine No. 3.

properties of this last 20 per cent were such that it could well be included in the hearts—that is, the separation of heads from hearts was not complete.

HEARTS.

The specific gravity and index of refraction of the first fraction of the hearts (fig. 33), indicated that the separation of the heads from the hearts was complete, but the boiling point, the odor and color of this first fraction indicated that a small amount of heads material was present in the hearts. It is probable that this incomplete separation

between the heads and hearts was largely due to the variation in the speed of the heads; this speed varied from 3.1 to 2.3 gallons per hour, and while the heads were running at 3.1 gallons per hour, some hearts material would be distilled over with the heads; when the speed dropped to 2.3 gallons per hour some heads material would escape in the returns from column A_1 to column A_2 and thus get into the hearts. The hearts also contained probably more heavy material with boiling points above 180° C. than is desirable in a well-refined turpentine.

TAILS.

The tails (fig. 34) contain about 5 to 6 per cent of hearts material showing an incomplete separation of hearts from tails.

REFINING OF CRUDE TURPENTINE

NO. 4.

The turpentine plant from which crude turpentine No. 4 was obtained is operated in connection with a saw-mill cutting longleaf pine almost exclusively. The raw material for the distillation consists of the sawdust together with the waste slabs, edgings, and trimmings, the large-sized material being "hogged" or made into chips before it is conveyed to the turpentine plant. The steam distillation process is used, the maximum pressure in the retorts being 10 pounds.

ANALYSIS.

The analysis of this crude turpentine is given in figure 35. This crude is of simple composition in comparison with Nos. 1, 2, and 3, being composed of about 84 per cent of turpentine and 16 per cent pine oil, with no evidences of the presence of any dipentene, or of any heads, except that the odor of the first fraction was somewhat different from that of the next few fractions.

It was desired to obtain one refined steam distilled turpentine containing comparatively large amounts of pine oil in order that the technical properties of two turpentines similar in composition, except

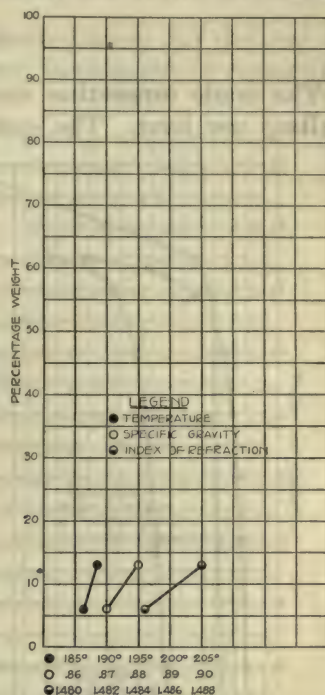


FIG. 34.—Boiling point, specific gravity, and index of refraction curves for "tails" obtained in refining of crude turpentine No. 3.

for the proportion of pine oil present, might be compared. It was decided therefore to separate this crude into about 2 per cent heads, 88 per cent hearts, and 10 per cent tails, and thus obtain a refined turpentine with more heavy oils than No. 2, the other steam distilled turpentine which was refined.

DISTILLATION.

The crude turpentine was run into the still at the rate of 24.8 gallons per hour. The speed of the heads varied from 0.2 to 1.0

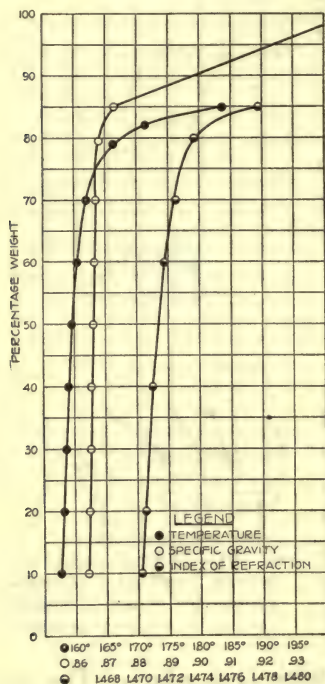


FIG. 35.—Boiling point, specific gravity, and index of refraction curves for crude turpentine No. 4.

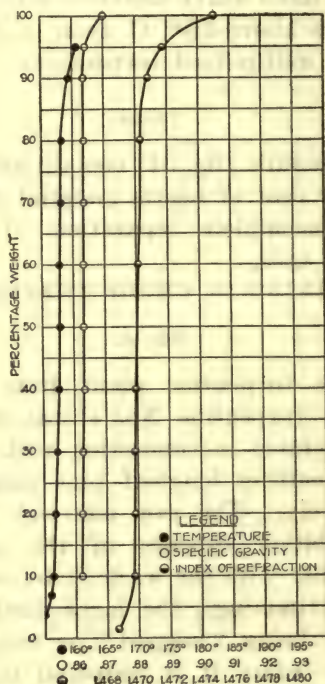


FIG. 36.—Boiling point, specific gravity, and index of refraction curves for "heads" obtained in refining of crude turpentine No. 4.

gallon per hour with an average of about 0.5 gallon per hour, and the hearts varied from 20 to 23 gallons per hour with an average of about 21.7 gallons per hour.

PRODUCTS.

HEADS.

The analysis of the heads from this run (fig. 36) showed the presence of only a very small amount of material that differed from the main portion of the turpentine. The first fraction of 1.5 per cent had a slightly lower index of refraction than any of the rest, and the

odor of the first 10 per cent of the distillate was quite different from the remainder. As in the case of crude turpentine No. 2, the amount of this material is so small, and its properties are so slightly different that it is unnecessary to attempt to separate it.

HEARTS.

The analysis of the hearts (fig. 37) showed about 6.5 per cent of pine oil present as was to be expected from the manner of regulating the distillation.

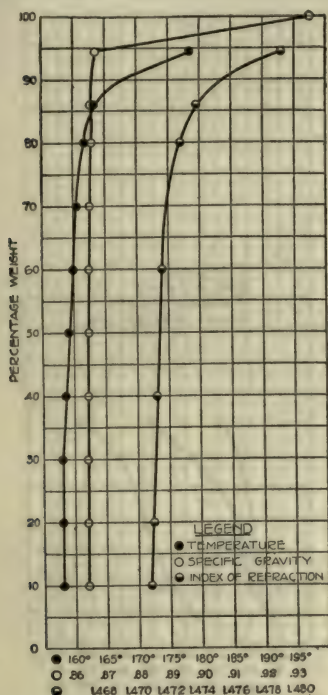


FIG. 37.—Boiling point, specific gravity, and index of refraction curves for "hearts" obtained in refining of crude turpentine No. 4.

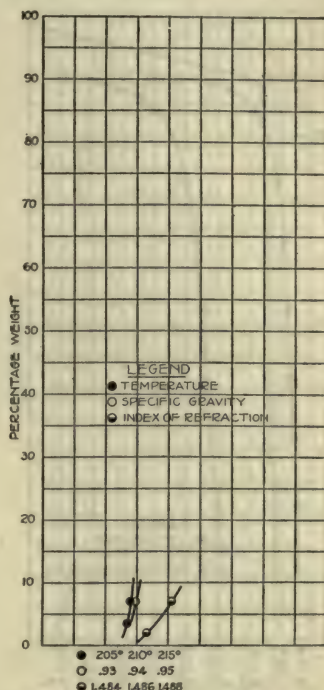


FIG. 38.—Boiling point, specific gravity, and index of refraction curves for "tails" obtained in refining of crude turpentine No. 4.

TAILS.

The tails (fig. 38) were apparently entirely free from hearts material, the very high values for the physical properties of the first fraction, 6.5 per cent, indicating that only a very small proportion of this fraction could possibly be made up of turpentine material. The separation in this case is therefore very satisfactory, since the hearts contain probably no more heavy material than is allowable in a good grade of turpentine, and the tails are practically free from turpentine.

PRODUCTS OBTAINED BY COMMERCIAL REFINING.

In order to obtain a comparison between the efficiency (as regards completeness of separation) of the column still, which was used in

this investigation, and that of the ordinary pot still commonly used in commercial practice, samples of the turpentine and pine oil, refined at the plant where this crude turpentine was produced, were obtained and analyzed. The samples were both from the same charge of crude and were refined by a single distillation with steam. The turpentine (fig. 39) is seen to contain about 12 per cent of heavy oils, while the pine oil (fig. 40) contains about 30 per cent of turpentine, showing that a very incomplete separation was obtained.

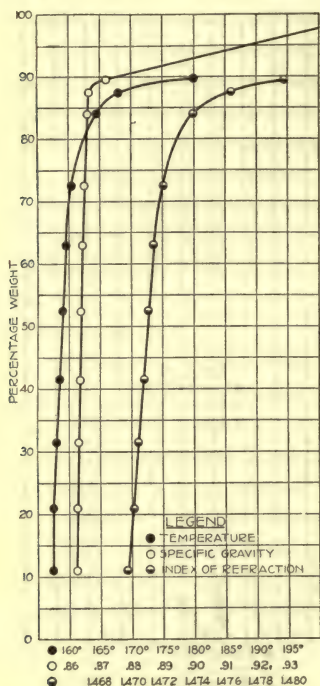


FIG. 39.—Boiling point, specific gravity, and index of refraction curves for a commercial wood turpentine (crude No. 4) refined in an ordinary pot still.

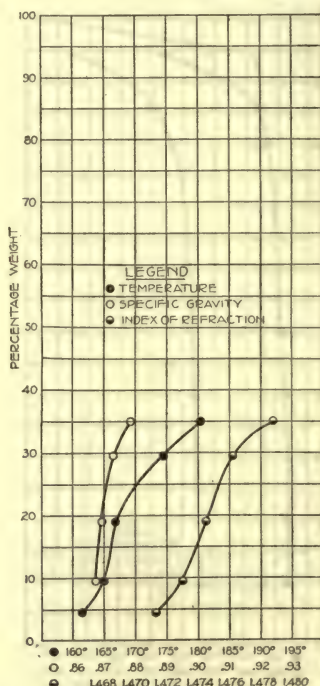


FIG. 40.—Boiling point, specific gravity, and index of refraction curves for commercial pine oil refined in an ordinary pot still.

REFINING OF CRUDE TURPENTINE NO. 5.

Crude turpentine No. 5 was produced from "lightwood" by means of the bath process of distillation. The "lightwood" in cordwood form is loaded on cars and these are run into large horizontal cylindrical retorts; then the hot bath material, pine-tar pitch, is circulated through the retort, the "extractor" and the furnaces in the order given. In the furnaces the temperature of the bath material is kept up to a maximum of 195° C. Live steam is blown through the bath in the extractor to remove the volatile oils which are carried by the bath from the retort. These volatile oils constitute the crude turpentine used in the refining experiments.

ANALYSIS.

The analysis of this crude turpentine is given in figure 41. There was no evidence of any heads material in this crude, except that there was a slightly different odor in the first fraction, and apparently about 57 or 58 per cent could be obtained which would contain a very small amount of heavy oils. It was decided, therefore, to attempt to separate the crude into the smallest possible quantity—2 per cent—of heads, 55 per cent of hearts, and the rest tails.

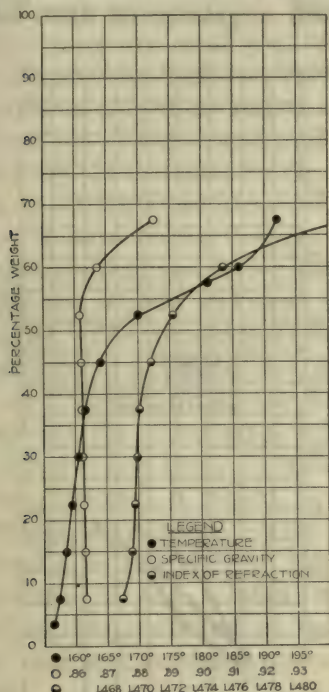


FIG. 41.—Boiling point, specific gravity, and index of refraction curves for crude turpentine No. 5.

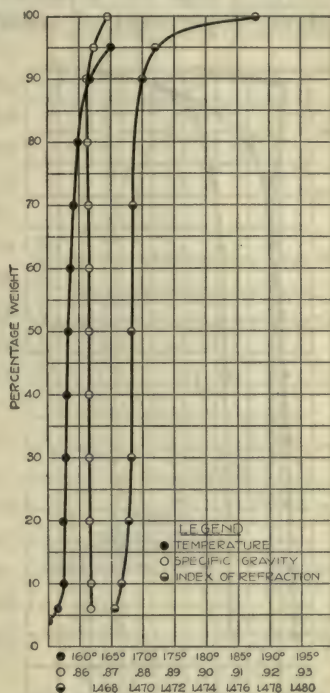


FIG. 42.—Boiling point, specific gravity, and index of refraction curves for "heads" obtained in refining of crude turpentine No. 5.

DISTILLATION.

The crude was run into the still at the rate of 25 gallons per hour, and the steam and water were adjusted to give as nearly as possible 0.5 gallon per hour of heads and 13.75 gallons per hour of hearts; these adjustments were maintained within the usual limits of variation.

PRODUCTS.

HEADS.

The analysis of the heads thus obtained is given in figure 42. From the boiling point and index of refraction curves it is apparent that

the first 10 per cent of the distillate contains a small amount of material with properties somewhat different from the rest of the distillate, and this was further indicated by the odor of the first fractions. As with turpentine No. 2 and No. 4, however, it is probable that there is so little of this material present that it is not only impracticable but unnecessary to attempt its separation.

HEARTS.

The hearts (fig. 43) are seen to be almost free from heavy oils, only about 2 per cent of material with a gravity of 0.890 being

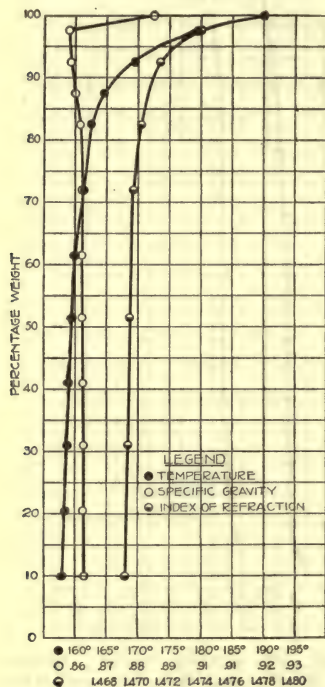


FIG. 43.—Boiling point, specific gravity, and index of refraction curves for "hearts" obtained in refining of crude turpentine No. 5.

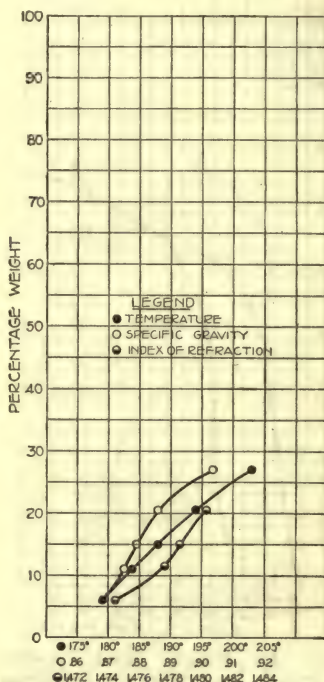


FIG. 44.—Boiling point, specific gravity, and index of refraction curves for "tails" obtained in refining of crude turpentine No. 5.

present, and the low gravity and index of the residue indicating that even this 2 per cent is largely composed of substances similar to the last few fractions of the distillate. A very nearly complete separation of hearts free from tails was therefore accomplished in this run.

TAILS.

The tails, however, are seen (fig. 44) to contain probably 15 per cent of hearts material, so that the separation of tails free from hearts was not complete.

REFINING ON A COMMERCIAL SCALE.

The refinings of the five crude turpentines were intended not only as experiments in refining, but also as studies of the composition of crude wood turpentines and the products obtainable from them. Especially, they proved to be a means of obtaining samples of wood turpentine with known compositions and methods of production, so that these samples might be used in comparative tests to determine the industrial value of wood turpentines of different composition. There was no aim to secure ideal conditions for the different distillations; throughout the experiments the arrangement of the still was not changed, and only in two cases were the distillations repeated in order to secure more accurate results. Hence, it must not be thought that, in all cases, the best results were obtained, or that the column was most suitably arranged. The composition of crude turpentines 2, 4, and 5, for instance, was such that it was not necessary to separate the constituents with boiling points below those of refined turpentine; instead, it would have been preferable to produce a refined turpentine and a refined pine oil, but the arrangement of the column was not suited to the production of these, and in no case was a refined pine oil produced.

However, in another piece of work under conditions more nearly commercial, the column was arranged for the production of a refined pine oil as well as a turpentine.

FIELD WORK ON A CRUDE TURPENTINE.

In this case the still was set up at the plant where crude turpentine No. 5 was produced. The arrangement of the still was similar to that in the experimental work, except that the crude reservoir was modified for continuous long runs, and the grouping of the sections was changed so that the upper column, A_1 , contained 13 boiling cap sections and 3 reflux condenser sections, while the lower column, A_2 , contained only 5 boiling cap sections and 2 reflux condenser sections. The refined turpentine was distilled from A_1 and

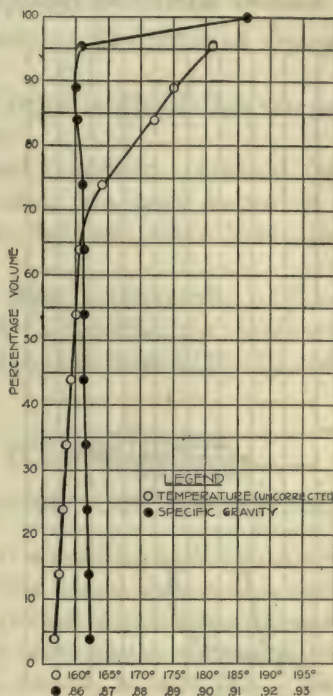


FIG. 45.—Boiling point and specific gravity curves for turpentine obtained in commercial refining of crude turpentine No. 5.

the refined pine oil from A_2 . (See fig. 20.) The analyses of the products obtained from one run, as shown in figures 45 and 46, give a good idea of the separations obtained under these conditions. The turpentine contained about 4 per cent pine oil, and the pine oil contained about 2 per cent turpentine.¹ This seemed to be the most complete separation possible, since with larger proportions of pine oil in the turpentine the amount of turpentine in the pine oil decreased, and vice versa.

Another interesting result was obtained in the test for determining the proper control of the distillation, in which it was found that the control of the still could be secured through the specific gravity of the products rather than through an arbitrary speed. Where the specific gravities of the two constituents to be separated are different, the specific gravity gives a nice control; but with a crude turpentine, such as No. 3, where two products (heads and hearts) have nearly identical gravities this method is not applicable.

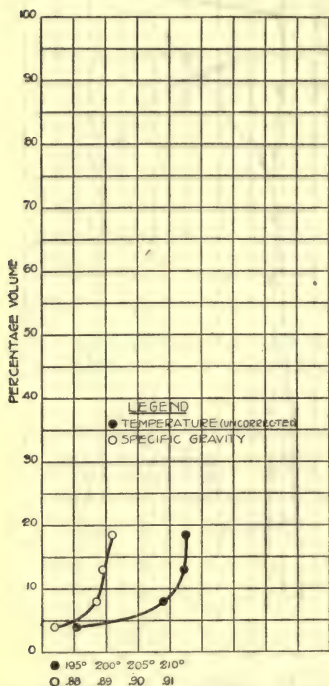


FIG. 46.—Boiling point and specific gravity curves for pine oil obtained in commercial refining of crude turpentine No. 5.

Group 2.—Includes crude turpentines 1 and 3, which were produced at temperatures above 200 C.

SUMMARY OF EXPERIMENTS.

The crude turpentines that were analyzed are sharply divided into two groups according to their compositions and to the methods used in their production. The means for their proper refining naturally follow the same grouping, which is as follows:

Group 1.—Includes crude turpentines 2, 4, and 5, which were produced at temperatures below 200° C.

¹ It will be noticed that figs. 45 and 46 are different in many ways from the preceding distillation curves. Index of refraction curves are not given, the boiling-point values are uncorrected, and the percentages are by volume and not by weight. These represent the results of analyses with portable apparatus, which can be set up and used without the conveniences of the chemical laboratory. Two 50 c. c. cylinders, graduated to 1 c. c., and one 500 c. c. measuring flask, take the place of the more delicate balances in determining the percentages of distillate, and the refractometer and barometer can be dispensed with; otherwise the method is the same. For many purposes this method is sufficient; but for close, careful work, the refinements described on pages 11–13 are necessary.

The following conclusions have been reached for the refining of crude wood turpentine like those of group 1:

(1) A fractional distillation without chemical treatment is sufficient for the production of good grades of turpentine and pine oil.

(2) The separation of light oils with boiling points lower than those of the turpentine is unnecessary.

(3) Better separation of the turpentine from the pine oil, or, in other words, a greater purity of the final products is obtained with a column still than with an ordinary pot still.

In the refining of crude wood turpentine like those of group 2 the following conclusions apply:

(1) A chemical treatment is required before a distillation can produce a good grade of refined turpentine.

(2) The color and odor of the refined turpentine obtained from the chemically treated crudes by distillation are much improved by the separation of the "light oils" with boiling points lower than those of the turpentine.

(3) Although no data are given to show what results might be obtained in an attempt to separate the "light oils" by distillation in an ordinary pot still, yet it is probable that these light oils could not be successfully separated.

COMPOSITION OF WOOD TURPENTINES.

THE RELATION BETWEEN PRODUCTION AND COMPOSITION.

If the crude turpentine are arranged according to the maximum temperatures of their production, the order would be 4, 2, 5, 1, and 3. The proportion of dipentene in the refined turpentine prepared from these crudes increases in the same order as the maximum temperatures of production. (See figs. 37, 38, 43, 14, and 33.) There would have been a more proper comparison between maximum temperatures and proportion of dipentene in the crude, rather than in the refined, turpentine, but the analyses of the crude turpentine do not in all cases indicate the proportion of dipentene present, as, for instance, in crude turpentine 1 and 3. (Figs. 21 and 30.)

As was to be expected, there is also a relation between maximum temperatures and the amount of "light oils" (with boiling points below 157° C.); for example, in crude turpentine 1 and 3, produced at (unknown) higher temperatures, light oils are present.

A similar relation holds between the maximum temperatures of production and the quantity of the products removable by treatment with caustic soda.

These relations between composition and temperature of production are given in Table 2.

TABLE 2.—*Relation between temperature of production and composition of crude wood turpentine.*

Crude turpentine.	Maximum temperature of production.	Dipentene in refined turpentine.	Light oils, boiling point below 157° C., insoluble in NaOH.	Oils, soluble in NaOH.
No.	° C.	Per cent.	Per cent.	
4	116	Practically none. ¹	None.	None.
2	148	About 10.	None.	None.
5	195	About 15.	None.	None.
1	High.	About 25.	About 3.	Some.
3	Higher.	About 40.	About 11.	More.

¹ For a more plausible reason than difference in temperature for the smaller amount of dipentene in turpentine No. 4, see page 53.

COMPARISON WITH GUM TURPENTINES.

A study of the variations in composition of gum turpentine just completed at the Forest Products Laboratory indicates that the volatile oil freshly distilled from oleoresin obtained by chipping live trees of longleaf pine contains only very small amounts of heavy oils (with gravity above 0.875). Therefore the 5 per cent of heavy oil shown in figure 2 is probably due to a partial oxidation of the terpenes in the original freshly distilled oil. Except for the presence of these heavy oils, figure 2 represents the composition of a typical gum turpentine, though in some the range of the values for the physical properties of the fractions might be slightly lower; in others, slightly higher.

HEAVY OILS.

On the other hand, the volatile oil obtained by the distillation of pitchy longleaf pine contains considerable quantities of heavy oils, and their presence in refined wood turpentine may result from incomplete separations of heavy oil from turpentine, even with an efficient column still. Still less perfect separations can be made with the pot stills commonly used, and there is a natural tendency on the part of the refiner to increase the quantity of refined turpentine by increasing the proportion of heavy oils in the refined product.

The usual presence of heavy oils in wood turpentine and their usual absence in gum turpentine is, then, the first difference between the two. Even in an old gum turpentine which contains heavy oils (like fig. 2), it is probable that the composition of these heavy oils is different from those in wood turpentine (see Composition of heavy oils, p. 58).

DIPENTENE.

The presence of dipentene in wood turpentine and its absence in gum turpentine is another difference. The refined turpentine ob-

tained from four of the five samples of crude contained dipentene; it is probable that most wood turpentine will contain appreciable amounts of this substance. In some 75 samples of gum turpentine which have been examined in the Forest Products Laboratory no dipentene has been detected by the method of analysis used. It appears that the presence of dipentene in wood turpentine is not entirely due to the temperature to which the turpentine is subjected, because most gum turpentine have been subjected to temperatures of at least 150° C. during their distillation, and yet crude turpentine No. 2, which was produced at 50 pounds steam pressure (148° C.), contained considerable quantities of dipentene. Dipentene was also found in the volatile oil distilled from lightwood by steam at atmospheric pressure.¹ The influence of temperature is indicated by the increased amounts of dipentene in wood turpentine produced at high temperatures. The dipentene in wood turpentine may, then, come from two sources, (1) from the dipentene present in the volatile oil as contained in the wood, and (2) from the transformation of some of the pinene into dipentene by the high temperatures used in the distillation.²

LIGHT OILS.

The presence of light oils in the wood turpentine produced at higher temperatures is the third possible difference. A crude turpentine containing light oils may be so refined that the product will be free from light oils (as in the case of crude No. 1), but the separation is difficult and can not readily be made without some kind of a fractionating column. If light oils are present in the crude turpentine small quantities are likely to be in the refined turpentine.

ROSIN OILS.

The fourth possible difference between wood and gum turpentine is difficult to describe, since it does not greatly affect the physical properties expressed in the distillation curves. For instance, in the analysis of refined turpentine No. 3 (fig. 33) it was quite noticeable that the odor and color of all the fractions differed from similar fractions of other turpentine. Except for the small amount of light oils present (see p. 42) the peculiar odor and color apparently did not tend to concentrate either in the first or last fractions, but were distributed rather evenly throughout the distillate. These properties

¹ "The Distillation of Resinous Wood by Saturated Steam," Bulletin 109, Forest Service.

² The absence of noticeable amounts of dipentene in turpentine No. 4 may be explained from its source—green sawmill waste; that is, old, dead, lightwood or stumps may contain dipentene while green freshly cut timber may not. It is expected that these debatable points will be settled by some work now under way in the Forest Products Laboratory.

were therefore probably due to the presence of some materials other than terpenes, with boiling point, gravity, and index of refraction not much different from the terpenes; the only strong indication of the presence of materials other than terpenes was in the high index of refraction of the first 30 per cent. From the method of production of this turpentine, these other materials can only be high-temperature decomposition products of wood fiber or of rosin. Decomposition products of wood fiber could not be present in very appreciable proportion, because the oils from this source, which would be insoluble in caustic soda, with boiling points 155° to 180° , and gravity in the vicinity of 0.85 to 0.87, are extremely small in amount. The destructive distillation of rosin, however, will yield yellow oils answering these requirements; the odor of these rosin oils (rosin spirits, rosin naphtha) is very similar to the fractions of this destructively distilled wood turpentine, and it is likely that the peculiar odor and color of destructive wood turpentines are due largely to the presence of "rosin spirits."

These comparisons of composition show that most wood turpentines will be sufficiently different in composition from gum turpentines, so that the method of analysis used in these investigations will readily distinguish between them; a wood turpentine produced from green timber at such low temperatures that no dipentene is formed and so refined that no heavy oils are present is, however, very similar to a gum turpentine; that is, the terpenes present in the wood from freshly cut long-leaf pine are very similar to the terpenes in the oleoresin obtained by chipping the live tree. The boiling point, the specific gravity, and the index of refraction curves from the two turpentines do not differ greatly, but these properties seem to be slightly lower in the fractions from the wood turpentines (compare figs. 2 and 37). This difference is, however, not great enough to provide a sure means of distinguishing between the two kinds of turpentines, since a gum turpentine with low values might be almost the same as a wood turpentine with high values. It is probable, therefore, that the same constituents are present in each, but that more of the higher boiling and higher gravity terpenes are generally present in the gum turpentines.

ODOR.

There is a considerable difference in odor between the gum turpentines and the wood turpentines, even between those which are nearest alike in composition. In most cases, however, this difference in odor is not due to differences in the main constituents. For instance, in the refined turpentine from No. 4 the main constituents are probably the same as in the gum turpentine shown in figure 2, and yet there is a decided difference in the odor of these two turpentines. Even the

presence of dipentene does not fully account for the difference in odor between wood and gum turpentine. Refined turpentine 2 and 5, both of which contained considerable quantities of dipentene, resembled gum turpentine in odor more than the products from No. 4, which was more nearly like gum turpentine in composition. It seems likely, therefore, that the distinctive odor of many wood turpentine is due to some material which occurs in so small a quantity that its presence is not readily indicated in any other manner than by the odor. The odor of a wood turpentine like No. 3 is probably due to the presence of rosin spirits, as mentioned on page 53.

EFFECT OF COMPOSITION ON TECHNICAL PROPERTIES.

The most valued properties of turpentine are usually considered to be its volatility, its solvent power, and its "drying" properties.

VOLATILITY.

The volatility of wood turpentine as compared to gum turpentine would probably not be greatly affected by the differences in composition. This property is measured, approximately, by the range of boiling points of the various constituents. Large quantities of heavy oils decrease volatility, but it is doubtful whether the effect of small quantities, up to 10 per cent, would be noticeable in practice.

SOLVENT PROPERTIES.

It is likely that the solvent properties of dipentene are quite similar to those of pinene, since these terpenes are so much alike in other respects; the solvent properties of the other materials which do not occur in gum turpentine, but which may be present in wood turpentine are probably sufficiently similar so that the small proportion of these which may be present does not appreciably affect the solvent properties of the mixture.

DRYING PROPERTIES.

In drying properties dipentene is apparently equal to pinene, since it has been found that the time required for drying a mixture of dipentene and linseed oil is the same as for drying a similar mixture of pinene and linseed oil.¹ In the case of the drying properties it is not likely that small amounts of rosin spirits, light oils, or even heavy oils, would greatly affect the industrial value of the turpentine. Until further data are available on the relation between the composition and industrial value of turpentine, the differences in composition between wood and gum turpentine should not be considered as necessary indications of corresponding differences in value.

¹This was determined by Edward Kremers, of the University of Wisconsin, in an investigation of terpenes as oxygen conveyors.

CHANGES IN COMPOSITION AFTER REFINING.

The analyses of refined turpentine were made within a few days after the refining. Afterwards these refined samples were kept for considerable periods, some in glass carboys, others in wooden barrels or iron drums, and further analyses were made from time to time to determine any changes in composition. Since the methods of storage were not the same the results are not accurately comparable, though they indicate the kinds and amounts of change which may take place during storage.

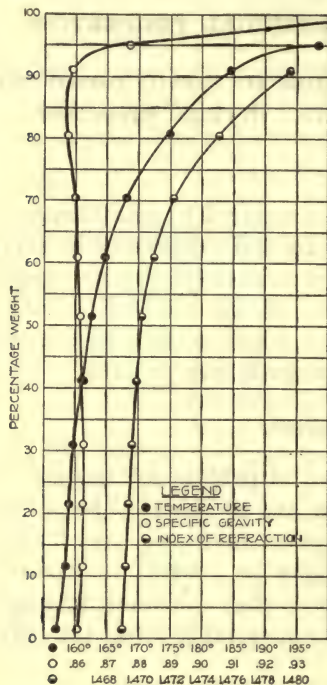


FIG. 47.—Boiling point, specific gravity, and index of refraction curves for the turpentine obtained from crude No. 1 analyzed after six months.

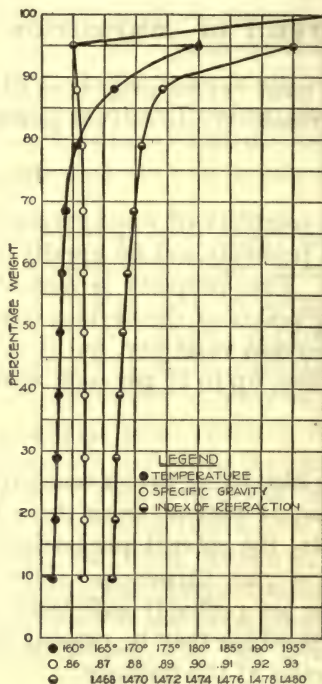


FIG. 48.—Boiling point, specific gravity, and index of refraction curves for the turpentine obtained from crude No. 2 analyzed after nine months.

Refined turpentine No. 1, the original analysis of which is shown in figure 24, was analyzed again after six months. (See fig. 47.) The only change which had taken place in this time was an increase in the amount of heavy residue from about 6 to 10 per cent; the odor and color had not been appreciably affected.

Refined turpentine No. 2 (original analysis, fig. 28) was analyzed after nine months (fig. 48); during this period the heavy residue had increased from 3.5 per cent with a gravity of 0.885 to 5.5 per

cent with a gravity of 0.94. Otherwise the sample seemed to be unchanged; it was still colorless and with no appreciable change in odor.

Refined turpentine No. 3 (original analysis, fig. 33) was analyzed after a period of nine months (fig. 49); there were no indications of change in composition.

Refined turpentine No. 4 (fig. 37) was analyzed after nine months (fig. 50); no apparent change in composition had taken place.

Refined turpentine No. 5 (fig. 43) was analyzed after 3 months

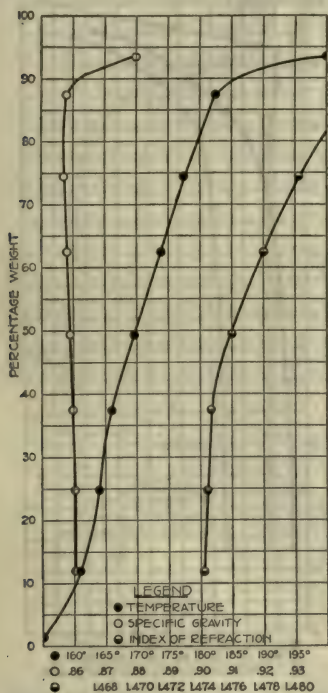


FIG. 49.—Boiling point, specific gravity, and index of refraction curves for the turpentine obtained from crude No. 3 analyzed after nine months.

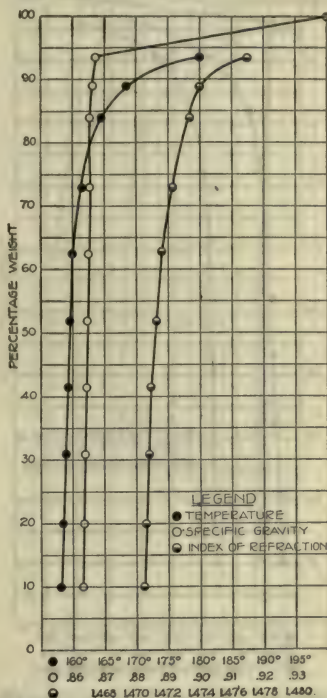


FIG. 50.—Boiling point, specific gravity, and index of refraction curves for the turpentine obtained from crude No. 4 analyzed after nine months.

(fig. 51); during this short time the amount of heavy residue had increased from 2 per cent with gravity of 0.885 to 4 per cent with gravity of 0.92. Other analyses, made after 6 and 10 months (figs. 52 and 53), showed a gradual increase in the amount of heavy residue. At the time of the last analysis a considerable yellow color had developed in this sample, possibly from the natural process of "aging," but more likely by contamination from an incompletely cleaned iron drum in which it was stored during a part of the time. In order to remove this color and also part of the heavy residue, the sample was

redistilled with steam in a small laboratory still without a fractionating column; the composition of the redistilled turpentine is shown in figure 54.

COMPOSITION OF HEAVY OILS IN WOOD TURPENTINES.

The presence of considerable quantities of heavy oils in the crude wood turpentine produced by steam distillation (see analyses of crude turpentine Nos. 2 and 4, figs. 26 and 35) indicates that some

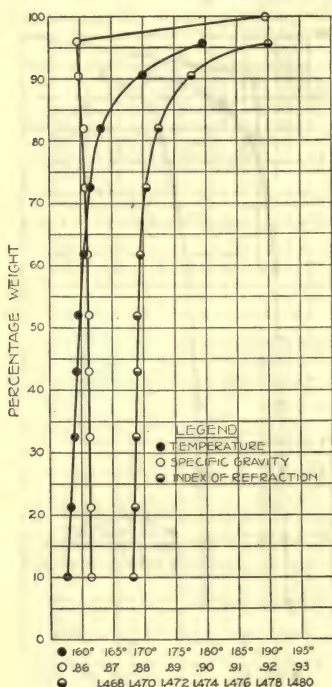


FIG. 51.—Boiling point, specific gravity, and index of refraction curves for the turpentine obtained from crude No. 4 analyzed after three months.

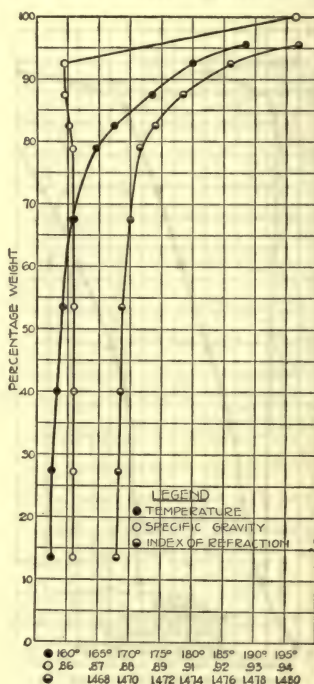


FIG. 52.—Boiling point, specific gravity, and index of refraction curves for the turpentine obtained from crude No. 5 analyzed after six months.

heavy oils, as such, are present in the wood itself, and are not formed during the distillation process. Experiments on steam distillation of longleaf pine have further confirmed these indications, since it was found that the proportion of heavy oil in the crude turpentine did not seem to be directly influenced by the temperatures of the distillation.

In the usual commercial refining practice these heavy oils are distilled over after the turpentine, and are marketed without further treatment as "pine oil." It was probably a sample of this pine oil

which was examined by Schimmel & Co.,¹ who identified the following bodies:

α -pinene, β -pinene (nopinene), camphene, limonene, dipentene, cineol, fenchyl alcohol, camphor, borneol, methyl chavicol, and α -terpineol. The first six of these, because of their low boiling points (all below 177° C.), are to be considered as normal constituents of the refined turpentine² which, however, occurred in the "pine oil" on account of incomplete separation in the refining.

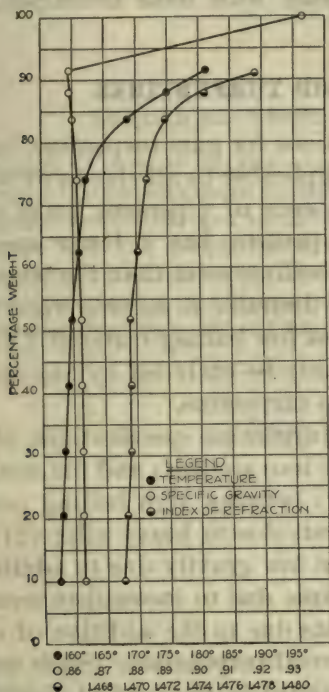


FIG. 53.—Boiling point, specific gravity, and index of refraction curves for the turpentine obtained from crude No. 5 analyzed after 10 months.

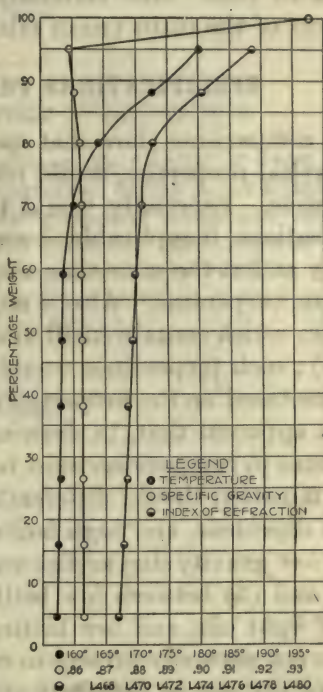


FIG. 54.—Boiling point, specific gravity, and index of refraction curves for turpentine obtained from crude No. 5 after redistillation.

The last five constituents have boiling points above 200° C. and may be considered as normal constituents of "pine oil" although,

¹ Semiannual Reports, Schimmel & Co., April, 1910, p. 100.

² With the exception of limonene and cineol, these low boiling constituents have been indicated as probable constituents of wood turpentine by the distillation curves. Because of the similarity between the two substances limonene could not be distinguished from dipentene by its effect on the distillation curves. Therefore limonene might have been present in any of the wood turpentines which contained dipentene. Cineol, however, has a considerably higher gravity (0.930) and a lower index of refraction (1.458) than dipentene and a boiling point (176° to 177° C.) which is near enough to that of dipentene so that the two would occur in the same fractions. It is possible, therefore, that very large quantities of cineol could not have been present in those turpentines whose distillation curves show a decreasing gravity and increasing index of refraction between 170° and 180°.

of course, small amounts of these substances may be present in the refined wood turpentine. (See p. 52.)

Since none of these five substances is known to be produced by the rapid oxidation of pinene or of the other terpenes in turpentine it is probable that the composition of the heavy oils in a wood turpentine (due to incomplete separation in refining) is not the same as those in a gum turpentine (produced by atmospheric oxidation). (See p. 52.)

The heavy oils in destructively distilled wood turpentines probably consist of these same materials, together with some decomposition products of the rosin (rosin oils).

SPECIFICATIONS FOR WOOD TURPENTINES.

The comparisons show that specifications for gum turpentine, based on physical properties, would not be applicable to all classes of wood turpentine. Especially would the presence of dipentene make such specifications inapplicable, because dipentene has a lower specific gravity and at the same time a higher boiling point than the terpenes in a gum turpentine. Also it might be desirable to include wood turpentines which contain small amounts of low boiling material (No. 3, fig. 33); such turpentines would certainly be excluded by any specifications based on the properties of gum turpentine.

It is apparent that, in drawing up turpentine specifications, these variations in composition must be taken into account; and the method of examination should differentiate (1) between high boiling points due to dipentene, and high boiling points due to heavy oils; (2) between low gravity due to dipentene and low gravity due to adulterations; and (3) between low boiling points due to incomplete separation of light oils, and low boiling points due to the addition of substances not normally present in crude wood turpentines. Such specifications can not be definitely recommended at the present time, because there is not sufficient information available on the technical value of wood turpentines of various compositions. Meanwhile, however, the method of analysis which is here presented will be of value in determining the composition of wood turpentines, and the various distillation curves will furnish, for comparisons, an idea of the variations possible in authentic wood turpentines.

APPENDIX.

PRACTICAL TESTS ON WOOD TURPENTINES.

Samples of each of the five wood turpentine, the refining of which is discussed in this bulletin, were submitted for practical testing to the Paint Manufacturers' Association and to several manufacturers of paints and varnishes. In these tests the sample turpentine were given the same numbers as the crudes from which they were distilled. The sample numbers and the numbers of the corresponding text figures, which show the composition of the turpentine at the time they were sent out for test, are as follows:

Turpen- tine No.	Figure showing com- position.	
	No.	Page.
1	47	56
2	48	56
3	49	57
4	50	57
5	54	59

The reports of the tests are given in full, as submitted by the various co-operators. There is no detailed analysis of the reports for the purpose of drawing conclusions in regard to the use of these turpentine in the paint and varnish industries. These can best be drawn by those thoroughly familiar with the requirements of these industries. However, the following significant facts are apparent:

(1) The lack of concordance between the reports shows the futility of attempting to establish a general relation between the composition and the industrial value of turpentine, without standard methods for determining the industrial value. The various individual reports probably indicate very accurately the relative industrial values of the different turpentine so far as the particular company making the report is concerned, but no general relation is established.

(2) Aside from the odor, the wood turpentine do not seem to vary much from each other or from gum turpentine; at least, the differences between the several turpentine, as indicated in any one report, are not greater than the differences reported for the same sample by different manufacturers.

(3) Although the odor is the property most emphasized, there is no unanimity of opinion as to the comparative odors of the different samples; in most cases

the objection is that the odor is different from gum turpentine rather than that it is inherently objectionable.

The reports follow:

REPORT 1.

Referring to the samples of turpentine which you sent us October 24, we have completed the preliminary investigation, and in carrying through the tests, which were made comparatively, we have added to the five samples submitted by you a sample of regular standard gum turpentine, marked A, and a sample of our standard wood turpentine which we are regularly using, marked B. We found the specific gravities to be as follows:

A, 0.8680.
No. 4, 0.8680.
B, 0.8650.
No. 3, 0.8650.
No. 1, 0.8650.
No. 2, 0.8635.
No. 5, 0.8625.

Flash point:

A and B, not taken.
No. 3, 113° F.
No. 1, 109.4° F.
No. 5, 105° F.
No. 4, 103.6° F.
No. 2, 101.8° F.

Staining power (on white paper, least staining power ranked first):

No. 5, first.
A, second.
No. 3, third.
B, fourth.
No. 2, fifth.
No. 1, sixth.
No. 4, sixth.

Evaporation (on white paper, shortest time ranked first):

No. 5, first.
A, second.
No. 2, second.
B, fourth.
No. 3, fourth.
No. 1, fourth.
No. 4, seventh.

Odor:

No. 5 and A, equal.
Nos. 1, 2, 4, and B, equal.
No. 3, objectionable odor and color.

When used for varnish reducing and carefully watched, we were unable to note any perceptible difference, either in setting or in completed drying after 24 hours. What we considered the most delicate test in practical use was in using them for thinning quick-drying black, and the results here were as follows (the time given is in minutes and represents the time when the black had dried sufficiently hard to be varnished over without rubbing up):

A	50 minutes.
B, 1, 2, 5	90 minutes.
3 and 4	105 minutes.

After six days the quick-drying black which had been thinned with these various turpentine was examined, and we found that a sample thinned with A showed little or no tendency to thicken up; No. 5 thickened slightly; and the balance had all thickened quite a little, but showed practically no differences.

In general way I would say that it is our judgment that for the general purposes for which material of this kind is used odor is the most important

feature, inasmuch as unfortunate odor has been the greatest objection to a large use of this class of material in the past, and at the present time it is the main cause of the prejudice against it.

From the standpoint of working qualities there is, as you will note, quite a decided action in slowing up the drying. I am not aware as to just what causes this, and of course for many purposes it would not be serious. There are many uses, however, for quick-drying color where the time requirements are so imperative that none of these materials could be used. We do not understand why the same measure of influence on the drying does not show up in connection with varnishes, but as a matter of fact it does not.

We should say that your sample No. 5 is decidedly better in general characteristics than any wood turpentine we have ever seen. In every point, excepting the drying in quick-drying black, it shows up entirely equal to straight turpentine.

Nos. 1, 2, 4 are satisfactory as wood turpentines, and would be usable for all purposes for which high-grade wood turpentine is at the present time usable.

The sample tested under B represents wood turpentine, of which we have used a considerable quantity and with which we have had practically no trouble excepting from the standpoint of odor.

It is impossible at the present time to substitute wood turpentine for a pure spirits in many places on account of the stronger odor, and from his standpoint your sample No. 5 looks particularly interesting.¹

REPORT 2.

Sample was taken from each can and compared in laboratory with pure gum spirits. The only objection raised was the decidedly objectionable odor in the No. 3 as well as the objectionable odor and slow evaporating in No. 5.

The samples were then tested as a thinner or solvent in an ordinary house paint and the same results obtained. The man applying the goods objected to the odor of both Nos. 3 and 5.

I then melted a batch of varnish and divided it into six equal parts, thinning five of these with the five samples of wood turpentine and the sixth part with pure gum spirits,² so that the body or base of all are identical.

After filtering and settling these six varnishes were submitted to practical tests by our head tester, who reports as follows:

Working with brush.....	Good.
Drying.....	{ Nos. 3 and 6 best.
	{ Nos. 1, 2, and 4 fair.
	{ No. 5 slow.

Color all up to standard. Odor: Nos. 3 and 5 strong odor; No. 3 most objectionable; Nos. 1, 2, and 4 fair; No. 6, sweet.

In having this test made we took great care both in making and testing the varnish. The man who tested did not know that he was not testing a regular run of six batches of one of our standard varnishes, which shows conclusively that he was doing his duty in an unbiased manner, and, as you will note, his report on odor corresponds with both laboratory report and test made by painter.

REPORT 3.

Material or subject.—Wood turpentine Nos. 2, 3, 4, and 5 from United States Department of Agriculture, Forest Products Laboratory.

¹The large variation in the drying properties found by the quick-drying black test indicated that this test offered a fairly simple method of determining the effect of the removal of the heavy oils on the drying properties. Small samples of each of the five turpentines were redistilled so as to prepare samples containing no heavy oils, and these special samples were submitted for the drying test. It was reported that the time of drying of all the samples had been brought closer together and closer to that of gum turpentine.

²Reported as No. 6.

Remarks.—For a comparative test with pure gum turpentine as to suitability for varnish making.

I. CHEMICAL TESTS.

	No. 2.	No. 3.	No. 4.	No. 5.	Gum turps.
Specific gravity.....	0.864	0.868	0.870	0.866	0.865
Residue on evaporation.....per cent..	.64	2.60	.79	.67	1.60
Iodine absorption, per cent.....	371.6	298.7	388.5	392.9	429

DISTILLATION TEST.

	Boiling point.				
	154° C.	155° C.	150° C.	153° C.	154° C.
165° C. per cent..	86	40	87	91	95
165° to 175° C., per cent.....	9	44	8	6	5
Over 175° C., per cent.....	5	16	5	3	
Total.....	100	100	100	100	100
Color.....	W. W.	Pale straw.	W. W.	Very pale straw.	
Odor.....	Best, mild.	Rank, penetrating.	Third, greasy.	Second, good but strong.	

¹ About.

II. *Varnish-making tests.*—Solvent power was tested by mixing 10 parts of each sample (also of gum turps.) with 1 part of a hard gum varnish. The pure turpentine precipitated the gum within one-half hour; No. 2 followed very shortly; then No. 5 (after about 6 hours). No. 4 held the varnish in solution, showing only a slight separation at the surface edges after a week; No. 3 held the varnish in solution perfectly for more than two months.

Other qualities were tested by reducing equal weights of the gum and oil from an agricultural varnish taken from the same batch, with equal volumes of the samples, as well as with pure turpentine, in such a way as to have all conditions as nearly as possible the same. The varnishes so made were not filtered, but were allowed to settle and age for three months. The percentage of volatile matter was then determined and the varnish was submitted to the usual tests of body, flowing, drying, brushing, etc.

The percentages of volatile matter were: For (I) varnish made with gum turps., 53.50; (II) No. 2, 54.87; (III) No. 3, 52.35; (IV) No. 4, 53.32; (V) No. 5, 54.10. The gum turp. varnish was the same in "body" as IV (as shown by the rate of rise of the air bubble in a narrow test tube of the varnish); II and V were both slightly lighter in body, as would be expected from the higher percentage of volatile matter; but on the other hand III was still thinner, though showing less volatile. This fact is doubtless due to the much greater solvent power of turpentine No. 3, which results in a much less viscous solution.

Varnish IV showed the poorest flowing qualities; V was slightly better; I and II (in order) still better; and III much the best. The drying test shows that the varnishes with lighter body dry faster and harder, though II, in drying faster than III, shows an exception. The relative order is II first, III, V, IV, I. From a consideration of factors involved it is evident that if all were brought to the same body II, V, and VI would dry at about the same rate, followed by III and I.

In brushing qualities varnish I was ranked first, V next, III next, followed by IV and II. This ranking denotes the comparative ease with which a smooth, glossy film of proper thickness can be obtained upon a filled board.

The results of the tests may then be summarized as follows:

The samples are all stronger solvents than gum turpentine, No. 3 being the strongest; No. 2 and No. 5 only slightly better than the gum spirits. If odor could be disregarded, No. 3 would probably be in all respects the best substitute for gum spirits and would be an improvement on it in some ways; but

No. 3 is too disagreeable in smell to be used, except in small proportions. The choice therefore lies between No. 2 and No. 5, with the majority of points in favor of the latter.

REPORT 4.

VARNISH TESTS MADE WITH WOOD TURPENTINES SUBMITTED BY UNITED STATES FOREST PRODUCTS LABORATORY.

Two grades of varnish were made up from each of the wood turpentines—rubbing varnish and spar varnish. The formulas used throughout in making up these varnishes are as follows:

RUBBING VARNISH.

By measure.		By weight.	
Gums.....	21.4	Gums.....	27.97
Oils.....	19.4	Oils.....	18.62
Turpentine..	59.2	Turpentine..	53.26
		Driers.....	.15
Total..	100.0	Total..	100.00

SPAR VARNISH.

By measure.		By weight.	
Gums.....	16.4	Gums.....	20.17
Oils.....	42.3	Oils.....	41.5
Turpentine..	41.3	Turpentine..	38.2
		Driers.....	.13
Total..	100.0	Total..	100.00

In the manufacture of the varnishes it was noted that Nos. 2 and 4 had the best odor while Nos. 1, 3, and 5, under heat, gave off strong odors, somewhat irritating to the eyes. By heating up the various turpentines alone to 100° and observing the odor the same result was obtained.

The varnishes which were manufactured from these turpentines were applied to oak panels 6 inches wide 12 inches high and 1 inch thick. After the panels had been properly coated, they were subjected to a series of tests, and the data obtained from these tests are also recorded in the following charts.

EXTERIOR VARNISH.

No.	1.....	2.....	3.....	4.....	5.....
Panel No.	I.....	II.....	III.....	IV.....	V.....
Number of coats ..	3.....	3.....	3.....	3.....	3.....
Odor.....	Fair; resembles gum turpentine but not quite as nutty; slight ethereal odor.	Excellent odor; as good as pure gum turpentine.	Extremely bad odor; resembles pyroligneous acid and acetone.	Fair; resembles turpentine.	Fair.
Action on eyes.....	None.....	None.....	Extremely irritating.	Very slight....	None.
Ease of application ..	Easy.....	Drys quickly; easy; pulls when dry.	Easy.....	Easy.....	Easy.
General working character.	Easy; good....	Easy; good....	Easier than I or II.	Good.....	Easy.
Color density ¹	A.....	B.....	D.....	D.....	C.....
Period of tackiness:					
A coat.....	3 hr. 30 min.	3 hr. 30 min.	2 hr. 50 min.	2 hr. 55 min.	2 hr. 55 min.
B coat.....	6 hr. 10 min.	6 hr. 10 min.	6 hr. 5 min.	5 hr. 35 min.	5 hr. 30 min.
C coat.....	6 hr. 00 min.	5 hr. 45 min.	5 hr. 35 min.	5 hr. 10 min.	5 hr. 15 min.
Period of hard drying.	Allowed 2 days between application of first and second coats. 4 days allowed between second and third coats.				
Body (rise of a bubble 2.5 cm. long in a tube 0.8 cm. diameter and 28 cm. long).	10 seconds (average of 3 trials).	11 seconds (4 trials).	7.6 seconds (3 trials).	10.7 seconds (7 trials).	11 seconds (4 trials).

¹A, B, C, and D indicate degrees of darkness, A being darkest and D lightest.

RUBBING VARNISH.

No.	1.	2.	3.	4.	5.
Panel No.	VI.	VII.	VIII.	IX.	X.
Number of coats.	3.	3.	3.	3.	3.
Odor	Fair; resembles gum turpentine but not quite as nutty; slight ethereal odor.	Excellent odor; as good as pure gum turpentine.	Extremely bad odor; resembles pyroligneous acid and acetone.	Fair; resembles turpentine.	Fair.
Action on eyes	None.	None.	Extremely irritating.	Very slight.	None.
Ease of application	Easy.	Works easily at first but dries quickly and pulls on rubbing.	Very easy; does not dry readily under brush.	Harder to rub than VIII but easier than VII.	Same as No. 2
General working character.	Good.	Good.	Good.	Good.	Good.
Color density ¹	A.	C.	D.	A.	B.
Period of tackiness:					
A coat.	1 hr. 35 min.	1 hr. 35 min.	1 hr. 20 min.	1 hr. 30 min.	1 hr. 20 min.
B coat.	1 hr. 30 min.	1 hr. 25 min.	1 hr. 25 min.	1 hr. 30 min.	1 hr. 30 min.
C coat.	2 hr. 30 min.	2 hr. 25 min.	2 hr. 20 min.	2 hr. 15 min.	2 hr. 10 min.
Period of hard drying.	(Allowed 2 days before applying second coat.				
Body (bubble 2.5 cm. long rises in tube 0.8 cm. diameter and 28 cm. long).	(Allowed 4 days before applying third coat.				
	8 seconds (5 trials).	9.3 seconds (5 trials).	6.7 seconds (5 trials).	9.8 seconds (7 trials).	9 seconds (6 trials).

¹A, B, C, and D indicate degrees of darkness, A being darkest and D lightest.

Temperature of laboratory during application of varnish: A coat, 90.5° F.; B coat, 93.2° F.; C coat, 86° F.

PAINT TESTS WITH WOOD TURPENTINES.

After the varnish test had been finished, the various turpentine were used to thin one standard paint formula, and this paint formula was applied to a series of panels to be placed on the southern test fence. The formula selected for the test consisted of 45 parts corroded white lead, 45 parts zinc oxide, 5 parts of asbestine, and 5 parts of whiting. This paint was divided into portions and thinned respectively with the five turpentine submitted for test. The paint formula itself was made up in condition resembling a ready mixed paint, as found upon the market, but containing nothing in the vehicle portion except linseed oil and drier. This formula was thinned in the proportion of 1 pint of turpentine to a gallon of paint for the priming coat, and one-half pint turpentine and one half gallon linseed oil for the second coat. The third coat was used without reduction. During the application of these paints the painter noted that No. 3 had a very objectionable odor, while Nos. 1, 2, and 4 had a very good odor, No. 2 probably being the best. In working and flattening properties, Nos. 1, 2, 4, and 5 gave the best results, and fully as good results as with the use of pure gum spirits. * * *

The evaporative values of these turpentine are satisfactory in every case, No. 3 evaporating more rapidly than the others, and none of them leaving any stain.

NOTE.—The panels painted with these test paints were exposed in September, 1910 (see Bulletin 30, Paint Manufacturers' Association), and the report of an inspection made in February, 1912, includes the following paragraph:

The panels which were painted with pigment formula 31 ground in pure linseed oil and reduced with various wood turpentine as thinners, indicated that the highest types of these materials were well suited to replace pure gum spirits of turpentine as a thinner for paints to be exposed out of doors. Wood turpentine No. 2 gave the whitest surface, while wood turpentine No. 5 gave the darkest.

REPORT 5.

No. 2: This is the best sample both in regard to odor and color and other qualities; it compares satisfactorily with gum spirits.

Nos. 4 and 5: These rank about the same from our point of view, being somewhat inferior to No. 2. No. 4 is lighter in color but No. 5 has less objectionable odor.

No. 1: This is next in quality to No. 4 and No. 5.

No. 3. This is the least desirable of the lot, having very disagreeable odor and being very dark in color. It also forms considerable residue when distilled with steam.

From a commercial point of view, we believe that No. 2 could be used to a considerable extent in the paint and varnish industries in place of gum spirits, but the odor of all the others would prevent their use except in a very small way.

REPORT 6.

(BY NAVY DEPARTMENT.)

(1) Tests have been concluded on four samples of wood turpentine (5 gallons each) submitted for testing purposes by the Director, United States Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, Wis.

(2) Results of physical tests:

The sample turpentines were marked Nos. 2, 3, 4, and 5, and were utilized in place of pure spirits of turpentine in four paints mixed according to the following formulas, each paint, being mixed with a different turpentine sample.

	Parts by weight.
White lead-----	9½
White zinc-----	5
Lampblack in oil-----	½
Raw linseed oil-----	3½
Test turpentine-----	½
Japan drier-----	¼

A similar number of paints (four) were mixed according to the above formula, using, however, pure spirits of turpentine instead of the test materials. These paints were considered as standards against which the paints containing the sample turpentines were to be tested. The paints were applied for the foregoing purpose to steel plates, and after the paints had dried the plates were exposed to the weather under date of October 12, 1911.

(3) It was noted that the paints containing the samples Nos. 2, 4, and 5 dried out in about the same time as the standard paints, while the No. 3 sample paint seemed to dry somewhat slower than the others. The plates were taken down for examination July 28, 1912, when it was found that the paints containing the sample turpentines were in excellent condition and quite equal to the standard paints in every respect.

(4) A second batch of paints were made up to practically the same formulas as previously used, with the exception that one half of the linseed oil was replaced with blown fish oil. These paints were applied on steel plates as before and were exposed October 16, 1911, to the weather in comparison to standard paints. These standard paints were mixed according to the same formula as the test turpentine paints, with the exception that pure spirits of turpentine was used. When examined July 29, 1912, it was found that all of the paints were in good condition, being about the same as the first batch of paints which were made up. It was noted that the fish-oil paints dried much slower than the first series of paints, which were made up with the linseed oil, the No. 3 sample paint being the last of the second series to dry.

(5) A third series of paints were now made up according to the following formulas, standard paints being also made up for comparison (pure spirits of turpentine):

	Parts by weight.		Parts by weight.
White lead (dry)-----	40	White lead (dry)-----	40
Sublimed white lead (dry)-----	60	Sublimed white lead (dry)-----	60
Zinc oxide (dry)-----	70	Zinc oxide (dry)-----	70
Asbestine (dry)-----	20	Asbestine (dry)-----	20
Barytes (dry)-----	10	Barytes (dry)-----	10
Linseed oil-----	32	Linseed oil-----	50
Toch drier-----	0.3	Toch drier-----	0.3
Test turpentine-----	50	Test turpentine-----	32

The paints made up according to the first formula dried very rapidly and quite flat. This was due to the large amount of thinners used. The first coat dried hard in one and one-half hours, and there was no appreciable difference in the drying time of any of the paints. The test turpentine paints and the standard paints were applied to steel plates and were exposed to the weather October 16, 1911, and examined July 28, 1912. It should be noted that the paints mixed according to the above two formulas were applied over two priming coats of red-lead paint.

(6) Examination of the paints showed the paint surface to be still intact but easily chalked up, though no signs of corrosion were apparent. The test turpentine paints were equal in all respects to the pure spirits of turpentine paints.

(7) The paints made up according to the second formula were applied, as in all cases, for exposure test against standard paints on steel plates, and these paints were exposed to the weather October 16, 1911. The drying of these paints was somewhat slower than the first formula paints of the third series, but not excessively, the first coats drying in three hours. The paints containing samples Nos. 2, 4, and 5 were somewhat more rapid than No. 3 paint. The results of the examination showed the paints to be in very good condition, chalking but slightly and protecting well the steel plates.

(8) A series of inside white paints were made with the test turpentines, compared to standard inside white paints containing pure spirits of turpentine. The following formula was used:

	Parts by weight.		Parts by weight.
"Ponolith"-----	60	Linseed oil-----	50
Zinc oxide-----	120	Manganese drier-----	2
China clay-----	20	Test turpentine-----	50

The inside white paints, made up according to the above formula, were applied in different parts of a large room at separate times, so that the effect of painting in a confined space might be determined; no ill experience by the painters, and the odors were not unpleasant.

(9) The chemical analysis of the various samples of wood turpentine submitted follows. The samples show on examination:

	Sample.			
	2	3	4	5
Flash (open tester).....° F.	104	112	104	104
Specific gravity at 60° F.....	0.8670	0.8614	0.8680	0.8644
Refractive index at 60° F.....	1.4703	1.4768	1.4718	1.4704
Residue on evaporation at 212° F., per cent.	2.90	1.66	1.38	1.62
Unpolymerized residue, sulphuric-acid test, after ½ hour standing.....inches.	2.5	1.7	2.5	3.3
Unpolymerized residue, after 1 hour stand- ing.....inches.	4.2	3.3	4.2	5.0
Color.....	Yellowish.	Greenish yellow.	Water white.	Yellowish.

None of the samples leave a spot when one drop is evaporated from filter papers.

When subjected to distillation, 86 per cent of sample No. 2 passes over between 309° and 330° F., 96 per cent having passed over when a temperature of 355° F. has been reached; 55 per cent of sample No. 3 passes over between 314° F. and 330° F., 96 per cent having passed over when a temperature of 362° F. has been reached; 91 per cent of sample No. 4 passes over between 309° F. and 330° F., 97 per cent having passed over when a temperature of 350° F. has been reached; 89 per cent of sample No. 5 passes over between 311° F. and 330° F., 97 per cent having passed over when a temperature of 350° F. has been reached.

All possess the characteristic odor of wood turpentine.

It will be noted from the above chemical analysis that the samples are not in conformity with the specifications for spirits of turpentine, 52-T-2, of December 30, 1911, in the distillation tests; all practically conform in specific gravity;

all conform to the sulphuric-acid tests and further conform to the refractive index test. Sample No. 4 is the only sample reported as "water white."

(10) In summing up the results obtained on the various tests to which the four samples of wood turpentine were subjected it is considered that they have proved to be entirely satisfactory for use in paints (both inside and outside paint) and that they are good grades of wood turpentine.

R. STOCKER.

[1st indorsement.]

1654

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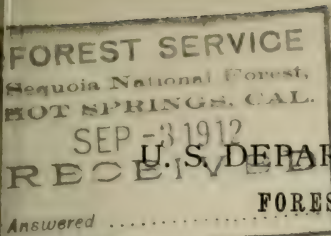
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Issued August 26, 1912.

U. S. DEPARTMENT OF AGRICULTURE,

FOREST SERVICE—BULLETIN 109.

HENRY S. GRAVES, Forester.

FOREST PRODUCTS LABORATORY SERIES.

DISTILLATION OF RESINOUS WOOD BY SATURATED STEAM.

BY

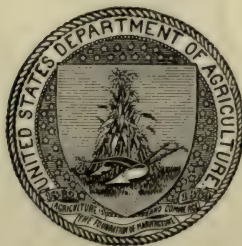
L. F. HAWLEY,

Chemist in Forest Products,

AND

R. C. PALMER,

Assistant Chemical Engineer in Forest Products.



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HENRY S. GRAVES, *Forester.*

ALBERT F. POTTER, *Associate Forester.*

HERBERT A. SMITH, *Editor.*

BRANCH OF PRODUCTS.

MCGARVEY CLINE, *Director, Forest Products Laboratory, in charge.*

HOWARD F. WEISS, *Assistant Director.*

WOOD DISTILLATION.

L. F. HAWLEY, *Chemist in Forest Products, in charge.*

E. BATEMAN, A. W. SCHORGER, *Chemists in Forest Products.*

F. W. KRESSMANN, R. C. PALMER, JACOB M. JOHLIN, JR., *Assistant Chemists in Forest Products.*

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE,
Washington, D. C., March 15, 1912.

SIR: I have the honor to transmit herewith a manuscript entitled "Distillation of Resinous Wood by Saturated Steam," by L. F. Hawley, chemist in forest products, and R. C. Palmer, assistant chemical engineer in forest products, and to recommend its publication as Bulletin 109 of the Forest Service.

The investigation was carried on at the Forest Products Laboratory, maintained in cooperation with the University of Wisconsin.

Respectfully,

HENRY S. GRAVES,
Forester.

Hon. JAMES WILSON,
Secretary of Agriculture.

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RECEIVED

OCT 30 1914

Division of Forestry
University of California

DISTILLATION OF RESINOUS WOOD BY SATURATED STEAM.

PURPOSE OF THE STUDY.

The steam distillation process for obtaining volatile oils from the wood of longleaf pine has been the basis of a small industry since about 1903. Apparently the process was introduced in an attempt to produce wood turpentine at temperatures lower than those used in destructive distillation, and thus to obtain a product uncontaminated by the decomposition products of wood and rosin. Quite a large number of plants have been built to use either sawmill waste or lightwood, or both, but many have been abandoned, and probably not more than 15 were in operation in 1911. The quality of the crude turpentine produced usually has been very good, but because this is the only product obtained, or because the yield of this product is generally lower than that of "crude turpentine" from other processes, the plants have been successful only under especially favorable conditions.

The process seems to be very promising, however, when combined with others for the utilization of the steamed chips, as, for instance, for the extraction of the rosin with volatile solvents. The process might be favored, also, where the material would be largely used as fuel, or wasted, or where it is very cheap, or so poor in quality that more complicated processes would not be profitable. These conditions generally obtain with the waste wood of sawmills now used as fuel at the plant or burned on the rubbish pile. In these fields the steam distillation of resinous woods will undoubtedly expand.

There has been no uniformity in commercial practice nor in the opinions of the various operators as to the proper steam pressure, size of chips, or rapidity of distillation, and no experimental data have been published on the effects produced by these various, readily controlled variables. In methods described in patent specifications the greatest stress has been laid on the mechanical features of charging and discharging and of distributing the steam throughout the retort, which, although of great importance in economy of operation, throw no light on other equally important features.

There seemed to be, therefore, a profitable field for investigations to determine the relations between the conditions under which the distillation is conducted, on the one hand, and, on the other, the amount and kind of products, and the readiness with which they are obtained. The results of the investigations are set forth to help promote the expansion of the steam distillation industry, and to increase in this way the utilization of a class of material which now is wasted.

THEORETICAL CONSIDERATIONS.

In the description of the experimental work and the discussion of the results it will be necessary to refer constantly to certain theoretical principles which apply to the distillation of volatile oils with steam, and, in order to make the future discussion clearer, these principles are briefly presented.

In order to simplify the deductions, the following assumptions are made in regard to the resinous material contained in the "lightwood" from the longleaf pine:¹

- (1) It is composed only of turpentine, pine oil,² and rosin;
- (2) The components are all simple substances completely soluble in one another;
- (3) None of the components is soluble in water;
- (4) The turpentine and pine oil are both volatile, but the turpentine has the lower boiling point;
- (5) Rosin is nonvolatile.

While these assumptions are not strictly true in all cases, none of them is sufficiently incorrect to seriously affect the conclusions.

The following deductions can be made as to distillation of the resinous materials, defined by these assumptions, with steam:³

- (1) There will be a separation of the two volatile constituents, with turpentine in greater proportion in the first part of the distillate and pine oil in the latter part.
- (2) Under normal pressure the temperature of the distillation will be slightly above 95° C. at the beginning, and will rise throughout

¹ M. Vezes (Bull. Soc. Chem., 29, 470-478, 1903) has given a very clear and complete discussion of the principles underlying the distillation of the oleoresin from the maritime pine of France. For the simplification of the discussion, the following assumptions were made:

- (1) That the oleoresin is composed only of essence (turpentine) and colophony (rosin);
- (2) That these components are both simple substances completely soluble in each other;
- (3) That neither is soluble in water; and
- (4) That rosin is nonvolatile.

In regard to the oleoresin obtained by chipping the live longleaf pine tree of the United States the same assumption can be made, but the oleoresin contained in the pitchy "lightwood" from this species has another component, the heavy, high-boiling "pine oil," which must be taken into consideration in the discussion of the steam distillation of such wood.

² A discussion of the occurrence of pine oil in the "lightwood" of longleaf pine is given in Forest Service Bulletin 105, "Wood Turpentines, Their Analysis, Refining, and Composition," by L. F. Hawley.

³ A detailed discussion of the methods used in making these deductions can not be given here, since a complete exposition of the laws governing the distillation of a four-component system like this would require too much space.

the distillation, never quite reaching $100^{\circ}\text{C}.$, however, as long as any of the turpentine or pine oil remains undistilled.

* (3) If the pressure is increased the temperature will be increased; the temperature depends upon the pressure and on the concentrations of the turpentine, pine oil, and rosin; it will, however, never reach the steam temperature corresponding to the pressures used as long as any turpentine or pine oil remains undistilled.

(4) The proportion of oil to water in the distillate will decrease as the distillation progresses; this proportion is influenced only by the composition of the oleoresin at various stages of the distillation.¹

In these deductions it is considered that the system is in complete equilibrium; under such conditions the behavior of oleoresin when distilled with steam can be foretold with considerable accuracy, but in the distillation of wood containing oleoresin there is a disturbing factor which necessitates investigation of several variables (p. 18). This disturbing factor is the difficulty of keeping a complete equilibrium between the oleoresin and the steam, since the wood surrounds the oleoresin and tends to keep the steam from coming in contact with it.

The effects of the size of chips and of the rapidity of distillation and (aside from temperature changes) the effects of steam pressure are due to the influence of these factors on the completeness of equilibrium between the oleoresin and the steam, rather than to their influence on the behavior of the oleoresin and steam when completely in equilibrium.

EXPERIMENTAL METHODS.

GENERAL PROCEDURE.

The general procedure was to distill charges of the same sized chips under different conditions, and of different sized chips under identical conditions, and to note carefully the variations in the results of the distillations. It was not practicable to make all the runs on exactly comparable material, because a large number of charges of equal resin content could not be secured, and because the material could not be kept without some loss of volatile oil by evaporation. The runs were therefore made in groups with comparable material in each group; the results from each group were made comparable to some extent with those from other groups by a comparison of the resin content of the different groups. This was readily accomplished by distilling the sawdust obtained in the preparation of the material for each group, as this sawdust was a good average sample of the whole group.

¹ A change in the pressure at which the distillation is carried on might change the proportion of water to oil in the distillate, but this change would be slight and there is not sufficient information available to decide even the direction of this change.

APPARATUS.

A sketch of the retort used for the distillations is shown in figure 1. This retort was designed for use in extraction of tannin from wood and bark, as well as for distillation experiments; it was therefore made of copper and had the closed steam coils *B*. The perforated plate *C* was designed to hold up the charge while the lower head of

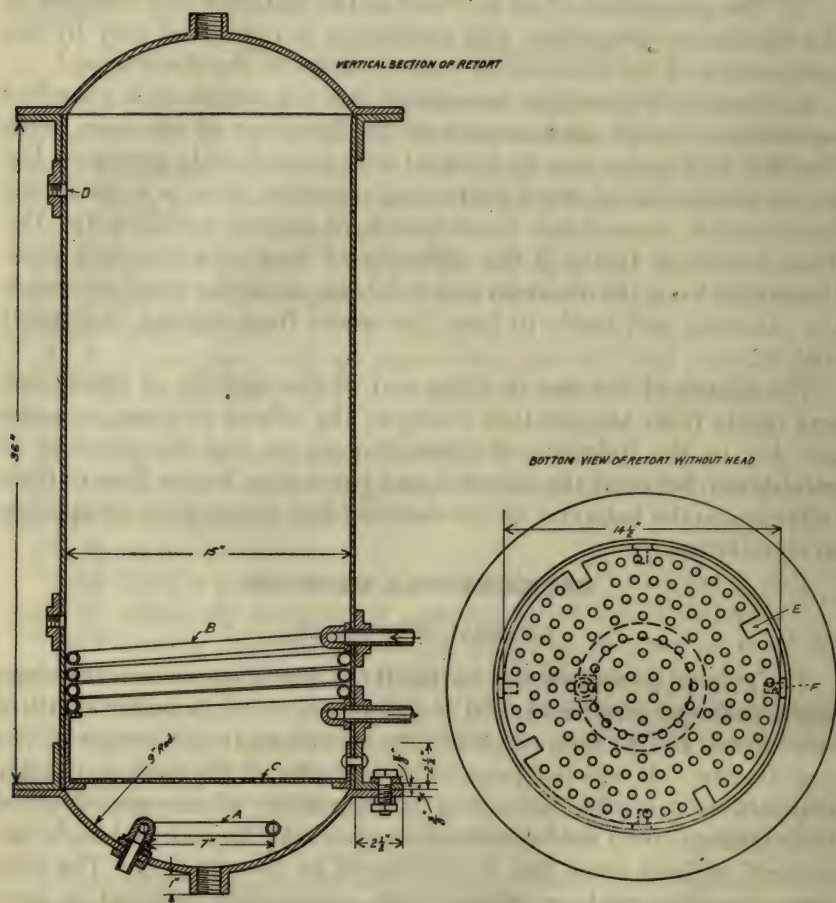


FIG. 1—Experimental retort.

the retort was taken off; then by revolving the plate until the slots *E* came opposite the supporting lugs *F*, the plate falls and the charge can be removed. The pressure gauge was attached at *D*. The other parts of the retort require no special description. An ordinary copper worm condenser was used and the distillate was caught in 1-liter graduated cylinders.

MATERIALS.

The J. G. Newman Lumber Co., of Hattiesburg, Miss., furnished for the experiments a large log of pitchy lightwood in which the pitch was distributed with unusual evenness. From this log it was possible to prepare a number of charges of chips, of which all from one portion of the log were similar in resin content.

PREPARATION OF MATERIAL.

The method of selecting material so that each charge should be comparable with the other charges in the same group is shown in Table 1 and figure 2. The sticks were sawed into sections at one time, but usually the sections were chipped only as required; that is, just before each distillation. Thus the sections for the last runs of a group had been longer cut than those of the first runs, and although they were kept in covered cans, or if without cover were piled close together, there was still some chance of loss by volatilization, especially when some time intervened, as in the large number of runs in Group I. To lessen the loss by volatilization during the chipping process, the chips were covered with water as fast as they were made. The sawdust charges were put into the retort without any delay after collection, except in runs 2 and 3, which were prepared and collected but not distilled at the same time with run 1.

DISTILLATION.

Only a brief description of the procedure needs be given here. The same general methods were used in all cases and the details are given later in the tabulated record of the runs.

REGULATION.

The speed and pressure of the distillations were regulated simply by the inlet valve at the bottom and by the outlet valve at the top of the retort. During distillation at atmospheric pressure the outlet valve was left open and the speed regulated by the inlet valve alone; but during distillation at higher pressure it was, of course, necessary to use both valves in order to keep both speed and pressure at the required values. The variation in speed was never more than one-half minute per liter of distillate, and in pressure never more than 2 pounds per square inch from the required values.

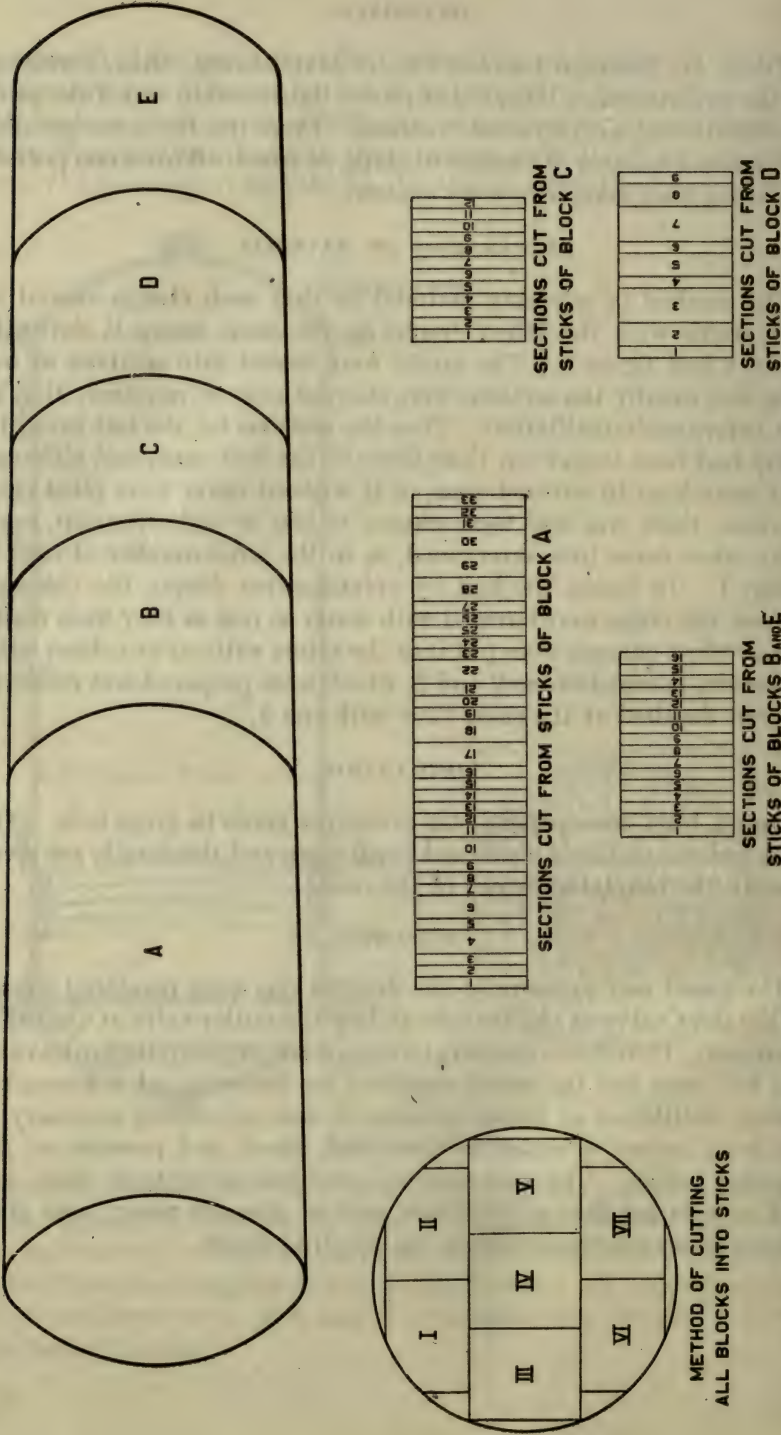


FIG. 2.—Diagram showing method of cutting up material. (In thickness the sections are drawn to scale, the smallest being 1 inch thick.)

TABLE 1.—*Experimental runs made and material used in each.*

Group No.	Run No.	Block.	Material used.			Remarks.
			Sticks.	Sections.	Size of chips.	
I	1	A			<i>Inches.</i> Sawdust.	Obtained in cutting sections. Do. Do.
	2	A			do.	
	3	A			do.	
	4	A	I to VII.	10, 22, 28.	1 x 1.	{ Chips from each section divided among the three runs.
	5	A	I to VII.	6, 18, 29.	$\frac{1}{2}$ x $\frac{1}{2}$.	
	6	A	I to VII.	4, 17, 30.	$\frac{3}{4}$ x $\frac{1}{2}$.	
	7	A	I to VII.	2, 14, 25.	$\frac{1}{4}$ x $\frac{1}{2}$.	
	8	A	I to VII.	3, 5, 9, 11.	$\frac{1}{4}$ x $\frac{1}{2}$.	
	9	A		13, 15, 19, 21.		
	10	A		23, 26, 27, 31.		
II	11	A			Sawdust.	Obtained in sawing slabs.
	12	A			1 x variable.	
III	13				6 x same.	{ One-half of each section used in each run.
	14	A	I to VII.	1, 7, 8, 12, 16.	$\frac{1}{2}$ of section.	
IV	15	A	I to VII.	20, 24, 32, 33.	$\frac{3}{4}$ of section.	{ Obtained in cutting sections.
	16	B			Sawdust.	
V	17	B	I to VII.	{ All odd numbers.	$\frac{1}{4}$ x $\frac{1}{2}$.	{ Chips from each section divided between each run.
	18	B	I to VII.			
	19	B	I to VII.	{ All even numbers.	$\frac{1}{2}$ x $\frac{1}{2}$.	Do.
	20	B	I to VII.			
	21	C			Sawdust.	Obtained in cutting sections.
	22	C	I, IV, VII.	1, 4, 7, 10.	2 x 2.	
			III, VI.	2, 5, 8, 11.		
			II, V.	3, 6, 9, 12.		
			III, VI.	1, 4, 7, 10.		
	23	C	II, V.	2, 5, 8, 11.	2 x 2.	
I, IV, VII.			3, 6, 9, 12.			
II, V.			1, 4, 7, 10.			
I, IV, VII.			2, 5, 8, 11.			
24		III, VI.	3, 6, 9, 12.	4 x 4.		
VI	25	D			Sawdust.	Obtained in cutting sections and in slabbing blocks.
	26	D	I to VIII.	1, 4, 6, 9.	1 x 1.	
	27	D	I to VIII.	2, 5, 8.	1 x 1.	
	28	D	I to VIII.	3, 7.	1 x 1.	
VII	29	E			Sawdust.	{ Chips from each section divided among the three runs.
	30	E	I, V.	1, 5, 9, 13.	Shavings.	
			II, VI.	4, 8, 12, 16.		
			III, VII.	3, 7, 11, 15.		
			IV.	2, 6, 10, 14.		
	31	E	I, V.	3, 7, 11, 15.	do.	
			II, VI.	2, 6, 10, 14.		
			III, VII.	1, 5, 9, 13.		
			IV.	4, 8, 12, 16.		
	32	E	II, V.	2, 6, 10, 14.	$\frac{1}{2}$ x $\frac{1}{2}$.	
			III, VI.	1, 5, 9, 13.		
			IV, VII.	4, 8, 12, 16.		
			I, V.	3, 7, 11, 15.		
	33	E	II, VI.	4, 8, 12, 16.	$\frac{3}{4}$ x $\frac{1}{2}$.	
			III, VII.	3, 7, 11, 15.		
			IV.	2, 6, 10, 14.		
I, V.			1, 5, 9, 13.			

DATA OBTAINED.

A typical data sheet which gives the records obtained in one distillation is shown in Table 2. The distillate was caught in 1-liter fractions and the time and pressure were recorded with every fraction. The amount of oil in each liter of distillate was determined as accurately as possible in the original receiver—a 1-liter cylinder graduated to 10 cubic centimeters; the oil was then separated from the water in a separatory funnel and its specific gravity determined. When the amount of oil in a single fraction was so small that the

specific gravity could not readily be determined, the oil was combined from a number of fractions. As a check, the combined oil from all the fractions, or from the fractions of certain portions of the distillation, was accurately measured.

Only the essential parts of the operations are presented, with computations derived from them. The discussion will be given after the experimental work has been described.

END POINT.

It will be noticed in Table 2 that the amount of oil in 1 liter of distillate progressively decreased, but this decrease was very slow toward the end of a distillation. It became necessary, therefore, to arbitrarily fix upon that ratio of oil to water which might be considered as the end of a distillation under a given set of conditions in order that the results of various distillations might be comparable and might be completed within practicable time limits. In the first five runs this end point was too high and too variable; when these runs were made it was not recognized that the end point must be carefully regulated. These first five runs are therefore not comparable with those which follow, in which the end point was kept at somewhat lower values. A charge distilled under one set of conditions until a certain end point was reached, if interrupted for an hour or more and then continued under the same conditions, produced a further supply of oil before the same end point was reached again. This additional oil amounted to from 2 per cent to 18.8 per cent of that which had been obtained before the distillation was interrupted. The greatest increases in yields due to this manipulation were in those runs in which there was still considerable oil present in the wood when the distillation was interrupted, although, of course, all the oil that could have been removed under the existing conditions had been extracted. For instance, in runs 14 and 15, after all possible oil had been distilled at 50 pounds pressure, interruption of the distillations overnight resulted in 18.8 per cent more oil in run 14 and 15.1 per cent more in run 15 under the same conditions; and in both these cases there was still considerable oil present in the wood, as shown by further distillation at increased pressures. In runs 11 and 21, however, after all possible oil had been distilled at atmospheric pressure, interruption of the distillation secured only 2.9 per cent and 4 per cent more under the same conditions; in these cases there were much smaller quantities of oil left in the wood than in runs 14 and 15.

After run 5 the proper end point for any distillation was considered to be reached only when the required ratio of oil to water in the distillate had been attained, both before and after interruption of the distillation.

TABLE 2.—*Typical data sheet, illustrating the records taken of the distillations.*

[Project 123—Run No. 23. Chips 1" x 2" x 2".]

Weight of can+water..... 160 lbs. Steam turned on..... 9.23
 Weight of can+water+chips..... 215 lbs. Distillate began to flow..... 9.28-30
 Weight of chips..... 55 lbs.

Time.	Distil- late.	Oil in distil- late.	Oil total com- puted.	Oil total deter- mined.	Specific gravity of oil.	Pressure.
a. m.	Liters.	c. c.	c. c.	c. c.		Pounds.
9.36	1	88	88		0.8810	70
9.48	2	154	242		.8794	72
9.59½	3	110	352	362	.8851	71
10.08	4	73	425			71
10.17	5	60	485		.8944	68
10.29½	6	55	540			71
10.40½	7	50	590		.8980	70
10.51½	8	44	634			72
11.09½	9	39	673		.8981	70
11.08½	10	24	697			69
11.18½	11	27	724		.9010	72
11.28	12	26	750			69
11.38	13	19	769			72
11.47½	14	22	791			68
11.57½	15	17	808		.9016	69
P. M.						
12.07	16	17	825	839		69
12.17	17	16	841			69
12.27½	18	16	857		.8987	72
12.38½	19	18	875			72
12.50	20	19	894			64
1.00	21	15	909			71
1.09	22	13	922		.8965	72
1.18½	23	11	933			71
1.27½	24	10	943			69
1.39	25	12	955	979		68
1.48	26	10	965			70
2.00	27	11	976			70
2.11½	28	10	986		.8958	68
2.23½	29	12	998			72
2.34½	30	8	1,006			73
2.45½	31	8	1,016	1,047		72
2.56½	32	10	1,026			63
3.07½	33	13	1,039			45
3.17	34	6	1,045			32
3.26½	35	5	1,050		.8936	20
3.37	36	4	1,054			12
3.43	37	1	1,055			4
3.45	38	1	1,056	1,095		0
A. M.						
9.23½	39	22	1,078			72
9.35	40	17	1,095		.8934	73
9.46	41	15	1,110			71
9.57	42	10	1,120			72
10.08	43	9	1,129			70
10.19	44	8	1,137		.9003	72
10.30	45	7	1,143			58
10.43	46	15	1,158			34
10.53	47	8	1,166			19
11.04	48	6	1,172		.8935	6
11.06	49	2	1,174	1,235		0

Distillation interrupted overnight.

No. of liters.	Yield per pound.	Effi- ciency.
	cc.	
16	15.3	0.95
25	17.8	.71
38	19.9	.51
49	22.4	.45

TABLE 3.—Summary of experimental runs.

Group No.	Run No.	Size of chip.	0 pounds pressure.		20 and 30 pounds pressure.		40 and 50 pounds pressure.	
			Yield per pound.	Efficiency.	Yield per pound.	Efficiency.	Yield per pound.	Efficiency.
		<i>Inches.</i>	<i>c. c.</i>		<i>c. c.</i>		<i>c. c.</i>	
I	1	Sawdust.....	20.6	1.71			c 9.9	0.45
	2	do.....					c 32.7	1.26
	3	do.....	12.5	1.56			c 8.2	.41
	4	2 x 1 x 1.....						
	5	2 x 1 x 1.....	6.4	.61	a 5.8	0.45	c 3.9	.33
	6	2 x 1 x 1.....	8.0	.55	a 5.1	.28	c 6.1	.24
	7	1 x 1 x 1.....	15.7	.63	a 4.6	.29	c 5.3	.24
	8	1 x 1 x 1.....	10.2	.46	a 6.5	.25	c 6.2	.19
	9	1 x 1 x 1.....			b 22.4	.46	d 3.0	.19
	10	1 x 1 x 1.....					d 27.2	.46
II	11	Sawdust.....	27.7	1.46			c 2.0	.25
	12	1 x variable.....						
	13	6 x variable.....						
III	14	1 x 5 x 8.....					d 19.7	.35
	15	1 x 1 x 1.....					d 24.5	.41
IV	16	Sawdust.....	25.2	.97			c 4.0	.31
	17	1 x 1 x 1.....			b 24.5	.50		
	18	1 x 1 x 1.....					d 26.8	.50
	19	1 x 1 x 1.....					d 25.3	.55
	20	1 x 1 x 1.....						
V	21	Sawdust.....	24.6	1.05			c 1.7	.23
	22	1 x 2 x 2.....						
	23	1 x 2 x 2.....						
	24	1 x 4 x 4.....						
VI	25	Sawdust.....	24.0	.89			c 2.6	.26
	26	1 x 1 x 1.....						
	27	2 x 1 x 1.....						
	28	3 x 1 x 1.....						
	29	Sawdust.....	19.3	.84			c 1.7	.17
			18.8	.94				
			.6					
	30	Shavings.....	19.4	.85			c 3.1	.39
VII	31	do.....	16.0	.84				
			1.4					
			17.4	.70			c 3.8	.38
	32	1 x 1 x 1.....						
	33	1 x 1 x 1.....						

a=20 pounds.

b=30 pounds.

c=40 pounds.

d=50 pounds.

TABLE 3.—Summary of experimental runs—Continued.

Group No.	Run No.	Size of chip.	70 pounds pressure.		Total pressure.		Speed per liter.	End point per liter.
			Yield per pound.	Efficiency.	Yield per pound.	Efficiency.		
I		<i>Inches.</i>	<i>c. c.</i>		<i>c. c.</i>		<i>Min.</i>	<i>c. c.</i>
	1	Sawdust.....			30.5	0.90	4	25 and 12
	2	do.....			32.7	1.26	4	18
	3	do.....			20.7	.74	4	17 and 12
	4	2 x 1 x 1.....	20.3	0.33	20.3	.33	5	12
	5	2 x 1 x 1.....	9.1	.33	25.2	.38	4	12
	6	2 x 2 x 2.....			19.2	.34	10	10
	7	1 x 1 x 1.....			25.6	.40	10	10
	8	1 x 2 x 2.....			23.0	.29	10	10
	9	1 x 2 x 2.....	4.5	.21	29.9	.34	10	10
	10	1 x 2 x 2.....	1.5	.12	28.7	.40	10	10
II	11	Sawdust.....			29.7	1.10	10	10
	12	1 x variable.....	25.7	.49	25.7	.49	10	10
	13	6 x variable.....	12.3	.29	12.3	.29	10	10
III	14	1 x 5 x 8.....	5.9	.17	25.6	.28	10	10
	15	1 x 1/2 x 1 1/2.....	4.7	.15	29.2	.32	10	10
IV	16	Sawdust.....			29.1	.75	10	10
	17	1 x 1 x 1.....					10	10
	18	1 x 1 x 1.....					10	10
	19	1 x 1 x 1.....					10	10
	20	1 x 1 x 2.....	29.8	.63	29.8	.63	10	10
V	21	Sawdust.....			26.3	.82	10	10
	22	1 x 2 x 2.....	23.1	.39	23.1	.39	10	10
	23	1 x 2 x 2.....	22.4	.46	22.4	.46	10	10
	24	1 x 4 x 4.....	23.6	.44	23.8	.44	10	10
VI	25	Sawdust.....			26.6	.70	10	10
	26	1 x 1 x 1.....	22.8	.45	22.8	.45	10	10
	27	2 x 1 x 1.....	21.4	.31	21.4	.31	10	10
	28	3 x 1 x 1.....	12.9	.29	12.9	.29	10	10
	29	Sawdust.....			21.0	.64	10	10
	30	Shavings.....					6 and 10	5 and 7
							10	7
					22.5	.73	6 and 10	5 and 7
VII	31	do.....					3	5
							10	7
							3 and 10	5 and 7
					21.2	.60	3 and 10	5 and 7
	32	1 x 1/2 x 1/2.....	20.1	.43			6	5
			.7				10	7
			20.8	.39	20.8	.39	6 and 10	5 and 7
	33	1 x 1/2 x 1/2.....	18.1	.39			3	5
			2.2				10	7
			20.3	.33	20.3	.33	3 and 10	5 and 7

DISCUSSION OF RESULTS.

Table 3 gives the conditions of the distillations and the results obtained. Under "Size of chip" the length parallel to the grain is given first. The probable error in the determinations of yields is apparently about 6 or 7 per cent and is due to difficulties in sampling, in regulating evaporation during the preparation of material, and in obtaining comparable end points in different distillations. An example of results which must be due to such errors is seen in runs 23 and 24. The chips in run 24 are larger than those in run 23, and the yield probably should be less, and certainly not greater, from the larger chips; yet the yields obtained from run 23 are 5 per cent less than those from run 24. A similar example is shown in runs 30 and 31. It might be thought that some of these variations in yields were due to incomplete distillation caused by the steam "channeling" through the charge in such a way that it never touched part of the wood; but in several runs, after all possible oil had been distilled under one set of conditions, the top of the retort was removed, the charge was well stirred, and the distillation continued under the same conditions as before, without any indications that the stirring had discovered undistilled material. It seems probable, therefore, that with a retort of the shape and size used the effect of incomplete distillation due to incomplete contact between the steam and the chips is negligible.

The values given under "Efficiency" are obtained by dividing the yields per pound of wood by the number of liters of total distillate, the efficiency factor being cubic centimeter of oil per pound of wood per liter of distillate, or
$$\frac{\text{c.c. oil}}{\text{pounds wood} \times \text{liters distillate}}.$$
 It might be thought that this "efficiency factor" would have more significance if it represented only the relation between oil and total distillate; but, as will be seen later,¹ this relation would be affected by the quantity of wood distilled. Of course the effect may not be in exact proportion to the quantity of wood, but it is probable that more nearly comparable efficiency factors are obtained by including it. These factors represent approximately the relative quantities of oil obtained in the various runs per unit of steam consumed, and of course exclude the steam which supplies the heat losses from other causes, such as radiation.

EFFECT OF SIZE OF CHIP ON YIELD AND EFFICIENCY.

In general, the smaller the chip the larger the yields and the higher the efficiency. This is shown in Table 4, which contains selected data from Table 3. Four groups of distillations are given, in each

¹ The reasoning given on page 19 regarding the effect of the size of retort on efficiency applies also to the effect of the amount of wood on efficiency.

of which all conditions except size of chips are, as nearly as possible, the same; in every case the smaller sized chips show the larger yield and higher efficiency. The effect on yield is not so marked in the case of runs 26 and 27 (and some of the other runs given in Table 3), but this is because the pressure was so high that nearly all the oil was removed even from the larger sized chips. Of two runs in which all the oil was removed even from the larger chips, the yields would, of course, be the same, but the efficiency would probably be higher with the smaller sized chips.

EFFECT OF PRESSURE ON YIELD AND EFFICIENCY.

In general, higher pressures give larger yields without lowering the efficiency. This is shown in Table 5, which gives three groups of runs, in each of which all conditions except steam pressure are as nearly similar as possible. In all cases the higher steam pressure produced the larger yield, with equal or greater efficiency. The effect of pressure on yields is also shown in another way in many of the runs in Table 3, in which, after obtaining all possible oil under one pressure, a further yield was obtained by continuing the distillation under a higher pressure.

EFFECT OF SPEED OF DISTILLATION ON YIELD AND EFFICIENCY.

Increased speed of distillation decreases both yield and efficiency. This is shown clearly in Table 6, which gives the results of two sets of two runs each, in both of which all conditions except speed were identical. It is probable that with the rapid passage of steam through the charge it is less completely saturated with oil vapors, and this directly decreases the efficiency. With this less complete saturation, the end point is reached sooner, and this, too, probably decreases the yield. This is indicated by the more nearly equal total yields obtained by finishing up the distillations at the same pressure but at lower speeds. However, the variation in efficiency is not proportional to the speed, since doubling the speed decreases the efficiency by only about 10 per cent—from 0.94 to 0.84 in runs 30 and 31 and from 0.43 to 0.39 in runs 32 and 33.

If, as seems probable, the effects of speed are due to the variations in the time during which the steam is in contact with the wood, then the size of the retort would have a similar effect; that is, a speed of 10 minutes per liter in a retort of a given size would be equivalent to 5 minutes per liter in a retort twice as large, since a unit of steam would be in contact with a unit of wood for the same length of time in either case.

TABLE 4.—*Effect of size of chip on yield and efficiency.*

Run.	Size of chip.	Pressure.	Yield.	Efficiency.	Speed per liter.	End point oil per liter of distillate.
	<i>Inches.</i>	<i>Pounds.</i>	<i>c. c. per lb.</i>		<i>Min.</i>	<i>c. c.</i>
7	1 x 1 x 1	(1)	15.7	0.63	10	10
8	1 x 1 x 1		10.2	.46		
12	1-inch sections from slab	70	25.7	.49	10	10
13	6-inch sections from same		12.3	.29		
14	1 x 5 x 8	50	19.7	.35	10	10
15	1 x 1 1/2 x 1 1/2		24.5	.41		
26	1 x 1 x 1	70	22.8	.45	10	10
27	2 x 1 x 1		21.4	.31		
28	3 x 1 x 1		12.9	.29		

¹ Atmospheric.TABLE 5.—*Effect of pressure on yield and efficiency.*

Run.	Size of chip.	Pressure.	Yield.	Efficiency.	Speed per liter.	End point oil per liter of distillate.
	<i>Inches.</i>	<i>Pounds.</i>	<i>c. c. per lb.</i>		<i>Min.</i>	<i>c. c.</i>
8	1 x 1 x 1	(1)	10.2	0.46	10	10
9	1 x 1 x 1		22.4	.46	10	10
10	1 x 1 x 1		27.2	.46	10	10
17	1 x 1 x 1	30	24.5	.50	10	10
18	1 x 1 x 1	50	26.8	.50	10	10
19	1 x 1 x 1	50	25.3	.55	10	10
20	1 x 1 x 1	70	29.8	.63	10	10

¹ Atmospheric.TABLE 6.—*Effect of speed on yield and efficiency.*

Run.	Size of material	Pressure.	Speed per liter.	End point oil per liter.	Yield.	Total yield.	Efficiency.	Total efficiency.
	<i>Inches.</i>	<i>Pounds.</i>	<i>Min.</i>	<i>c. c.</i>	<i>c. c. per lb.</i>	<i>c. c.</i>		
30	Shavings	(1)	6	5	18.8	18.8	0.94	0.94
		(1)	10	7	.6	19.4		.85
		40	10	10	3.1	22.5	.39	.73
31	do	(1)	3	5	16.0	16.0	.84	.84
		(1)	10	7	1.4	17.4		.70
		40	10	10	3.8	21.2	.38	.60
32	1 x 1/2 x 1/2	70	6	5	20.1	20.1	.43	.43
			10	7	.7	20.8		.39
33	1 x 1/2 x 1/2	70	3	5	18.1	18.1	.39	.39
			10	7	2.2	20.3		.33

¹ Atmospheric.

RELATIONS BETWEEN END POINT, YIELD, AND EFFICIENCY.

Table 2 shows that the amount of oil in a liter of total distillate is greatest at the beginning and decreases steadily as the distillation progresses, except when conditions are changed, and then the increase in the amount of oil per liter is usually only slight and temporary. This was true in all the distillations. It is evident, therefore, that the efficiency factor will decrease steadily throughout the distillation, and its final value will depend on the end point used. Thus, the efficiency can be increased by stopping the distillation before all the oil has been obtained, but this decreases the total yield of oil. On the other hand, the efficiency will be decreased by continuing the distillation until all possible oil has been obtained, although the total yield of oil is increased. For instance, in run No. 23 (Table 3), if the distillation had been stopped with an end point of 12 c. c. per liter, at the twenty-fifth liter the yield would have been only 15.3 c. c. per pound while the efficiency would have been 0.95. By continuing the distillation until the end point (after an interruption of the distillation) was 10 c. c. per liter, the yield was much larger, 22.4 c. c. per pound, but the efficiency was only 0.46.

THE PRESSURE REQUIRED TO DISTILL COMPLETELY DIFFERENT SIZES OF MATERIAL.

SAWDUST.

The volatile oil can not be completely distilled at atmospheric pressure even from a material as finely divided as sawdust. This can be seen from runs 11, 16, 21, 25, and 29, in which, after removing all possible oil at atmospheric pressure, a further distillation at 40 pounds pressure removed from 6.6 to 15.8 per cent more oil. After distillation at 40 pounds a further distillation at 70 pounds was without appreciable results. It can be safely stated that a pressure of 40 pounds is sufficient to remove all the volatile oil from material as small as sawdust. It is possible that lower pressures might give almost as good results, but this can not be determined from the data on hand.

CHIPS 1 INCH $\times$ $\frac{1}{4}$ INCH $\times$ $\frac{1}{8}$ INCH.

This size material can not be completely distilled at 30 pounds pressure (run 17) and probably not at 50 pounds pressure (run 18). In run 18, the yield obtained from chips 1 inch $\times$ $\frac{1}{4}$ inch $\times$ $\frac{1}{8}$ inch is almost the same, within the limit of possible variation, as from the sawdust of run 16, but apparently not quite all the oil has been removed.

CHIPS 1 INCH $\times$ $\frac{1}{2}$ INCH $\times$ $\frac{1}{2}$ INCH.

Chips of this size can not be completely distilled at 50 pounds pressure (run 19), but can at 70 pounds (runs 20, 32, and 33).

CHIPS LARGER THAN $1\text{ inch} \times \frac{1}{2}\text{ inch} \times \frac{1}{2}\text{ inch}$.

At the maximum pressure used, 70 pounds, chips larger than 1 inch $\times \frac{1}{2}$ inch $\times \frac{1}{2}$ inch can not be completely distilled, but as the size of chips is increased there is no sudden drop in the yields obtainable at this pressure until sizes larger than 2 inches with the grain (runs 27 and 28) ¹ and 4 by 4 inches across the grain are used (runs 14 and 24). It is probable that from 80 to 85 per cent of the oil could be removed from chips 2 inches $\times$ 4 inches $\times$ 4 inches by distillation at 70 pounds pressure.

EFFECT OF PRESSURE ON COMPOSITION OF OIL.

Analyses were made by the method described in Bulletin 105 ² of part or all of the oil from each of the runs, but there was so little difference between the various samples that all of the analyses need not be given, and only a few distillation curves, which show the main points of interest, are presented.

PINE OIL.

The proportion of pine oil in the crude turpentine did not vary, except in cases which could be explained by variation in other factors besides pressure, and therefore, so far as the results show, the pressure has no influence on the proportion of pine oil except the influence due to increasing the total yields. That is, the pine oil has a higher boiling point, and therefore would be removed last, giving a higher proportion of pine oil in those cases in which the yield is greater. In all cases where the total oil obtained was analyzed and where the oil was nearly completely removed from the wood the percentage of pine oil by weight varied only between 48 per cent and 52 per cent. ³ In cases where only part of the oil was removed by the distillation, as in run 4, the proportion of pine oil was less.

¹ In determining the percentage of total oil obtained in the runs of Group VI it must be remembered that the sawdust of run 25 was not exactly a representative sample of the material of that group, but that it was mixed with the sawdust obtained in cutting the slabs from the blocks. (See Table 1.) In several cases similar samples of sawdust obtained in cutting the slabs had been distilled and found to contain more volatile oil than the sawdust obtained in cutting the blocks. It is probable, therefore, that the proportion of volatile oil in the mixed sample of sawdust was somewhat greater than in the rest of the material in this group. This point is further indicated by a comparison of the yields obtained from the sawdust runs in the different groups. With the exception of Group VI (run 25) the yields from the sawdust decrease, as might be expected, for the reason that the material for the groups was cut from the same log in order of the group number, beginning at the butt end, and the content of volatile oil in the butt end was higher than in the upper portions of the log. It is probable, therefore, that a value of from 24 to 25 c. c. of oil per pound of wood would more nearly represent the volatile oil content of the group.

² Bulletin 105, Forest Service, U. S. Department of Agriculture, "Wood Turpentines, Their Analysis, Distillation, and Composition."

³ Exceptions were found to this in the oils from Group II, which contained about 28 per cent pine oil, but the material for this group did not represent a complete cross section of the log, being composed instead only of the outside pieces, the slabs. The outer layers of this log evidently contained a smaller proportion of pine oil than the rest of the wood.

DIPENTENE.

The detection of small differences in the proportion of dipentene present can not be made by the method of examination used, especially when such large proportions of pine oil are present. There seemed to be, however, more dipentene in the crude turpentines pro-

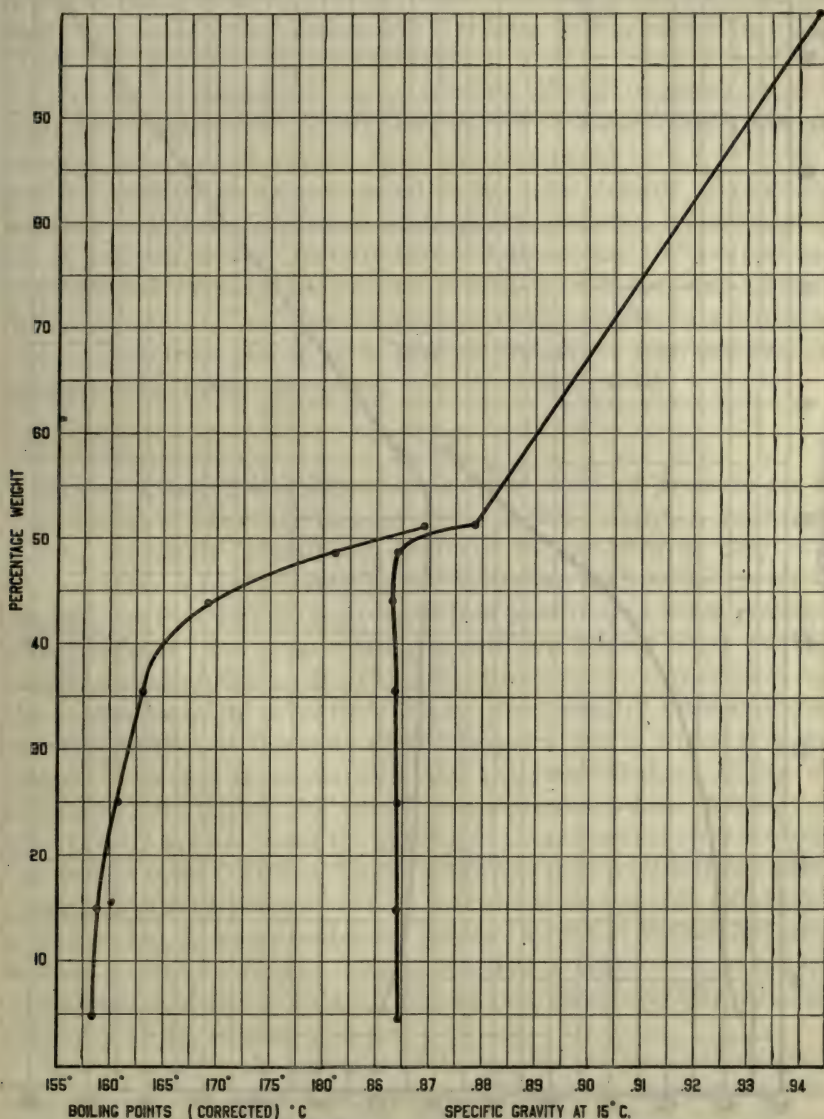


FIG. 3.—Boiling-point and specific-gravity curves for oil from run 21.

duced at higher pressures. Figures 3 and 4, representing the distillation curves obtained in the analyses of the oils from runs 21 and 23, respectively, illustrate this point. The oil obtained mostly at atmospheric pressure from sawdust (fig. 3) apparently contains less

dipentene than the oil distilled entirely at 70 pounds pressure (fig. 4); in the latter case the specific gravity values are lower and the proportion of the oil boiling between 165° and 180° is larger. Formerly it



FIG. 4.—Boiling-point and specific-gravity curves for oil from run 23.

was thought that the dipentene in wood turpentine was caused by the temperature used in distilling the oil from the wood, but indications of dipentene were found in all the samples of oil obtained in this

investigation, even in those produced at atmospheric pressure, and it is very probable that dipentene was present, as such, in the wood. In order to make sure that this material with low specific gravity and high boiling point was dipentene and not some other terpene with similar physical properties, a chemical examination was made of the fractions from 165° to 185° from some of the turpentines produced at atmospheric pressure and dipentene was identified by means of the tetrabromide, m. p., 125° – 126° .

In order to determine the possibility of the transformation of pinene into dipentene under the condition of steam distillation, the sawdust from run 29 was air dried and moistened with 1,175 c. c. of gum turpentine (an amount equal to the total volatile oil originally present) and then distilled at atmospheric pressure. The resultant oil, on analysis, showed no indications of dipentene. The experiment was repeated, making the distillation at 50 pounds pressure, but with the same result. These results preclude the possibility of a formation of dipentene from pinene at 50 pounds pressure or less, and indicate strongly that dipentene occurs, as such, in lightwood.

LIGHT OILS.

Figures 3 and 4 also illustrate another effect of pressure on the composition of the crude turpentines. In these analyses, as in many others, the crude turpentines produced at pressures as high as 70 pounds show a considerably higher value for the specific gravity of the first fraction than do the turpentines produced at lower pressures. This indicates that some substance with low boiling point and high gravity (above 0.870 at 15° C.) is produced at the higher pressures; this substance might come from the incipient stage of a decomposition of some portion of the resin at the temperature to which it is subjected. The first fractions from the analyses which contained this substance were slightly yellow and had a peculiar odor. A treatment with caustic soda reduced the gravity of these fractions but deepened the yellow color. It was found, however, that by the caustic-soda treatment of a turpentine such as that shown in figure 3, followed by a distillation, it was possible to prepare a refined turpentine which showed no abnormality of the first fraction in color, odor, or gravity. The presence of this substance should not, therefore, introduce any difficulty in the refining process.

Another test for the presence of decomposition products was made on several of the samples produced at different pressures by treating the oil with concentrated hydrochloric acid. A red color produced in this way is supposed to indicate the presence of rosin oil. There was only a very slight coloration of the oils produced at atmospheric pressure, but this coloration increased with the pressure, becoming very marked in the oils produced at 50 and at 70 pounds pressure.

FRACTIONATION OF THE OIL DURING DISTILLATION.

Some very interesting conclusions as to the manner in which the volatile oil leaves the wood can be obtained by comparing the values of the specific gravity of various portions of the distillate. As previously stated, the specific gravity of the oil was determined from each liter of distillate or from as many liters as were necessary to furnish the amount of oil required for a determination. Figures 5, 6, and 7 show these values of the specific gravity obtained in runs 16, 20, and 7, respectively, plotted against the percentages of the total oil obtained.

Figure 5 shows the changes in the specific gravity of the oil obtained during the distillation of a charge of sawdust, first at atmospheric pressure, and then at 40 pounds pressure. The first portions of the oil were nearly pure turpentine, but after about 44 per cent had been distilled the gravity increased rapidly, indicating the presence of pine oil in increasing quantities; when the distillation was from about 67 per cent to 83 per cent complete, the oil was nearly pure pine oil. The part of the curve up to 83 per cent resembles very closely a curve obtained from the steam distillation of a crude turpentine; that is, the presence or absence of the wood seems to have no effect on the manner in which the volatile oils are distilled. A difference is seen, however, in that portion of the curve beyond 83 per cent; after practically all possible oil has been removed by a continuous distillation at atmospheric pressure, interruption of the distillation, followed by a further distillation under the same pressure, produced a small further yield of oil with a lower gravity, and, on increasing the steam pressure, still more oil was obtained with a still lower gravity. This indicates that both the interruption of the distillation and the increase in steam pressure brought more oil into contact with the steam and that this oil contained some of the low-gravity turpentine material.

A very different behavior is shown in figure 6, which represents the distillation of chips 1 inch $\times$ $\frac{1}{2}$ inch $\times$ $\frac{1}{2}$ inch at a pressure of 70 pounds. In distillation under these conditions there was much less tendency for the oil to be separated as distilled, the gravity of the very first fraction being higher than that of pure turpentine and the gravity of the later fractions never reaching that of pure pine oil; that is, the turpentine and pine oil distilled together throughout the run. This indicates that new supplies of volatile oils were brought into contact with the steam more or less continuously throughout the distillation; had it been otherwise the turpentine would have distilled first and the last fractions would have been nearly pure pine oil.

A still more striking picture of the variation in the gravity of the distillate due to changes in the conditions of distillation is shown in

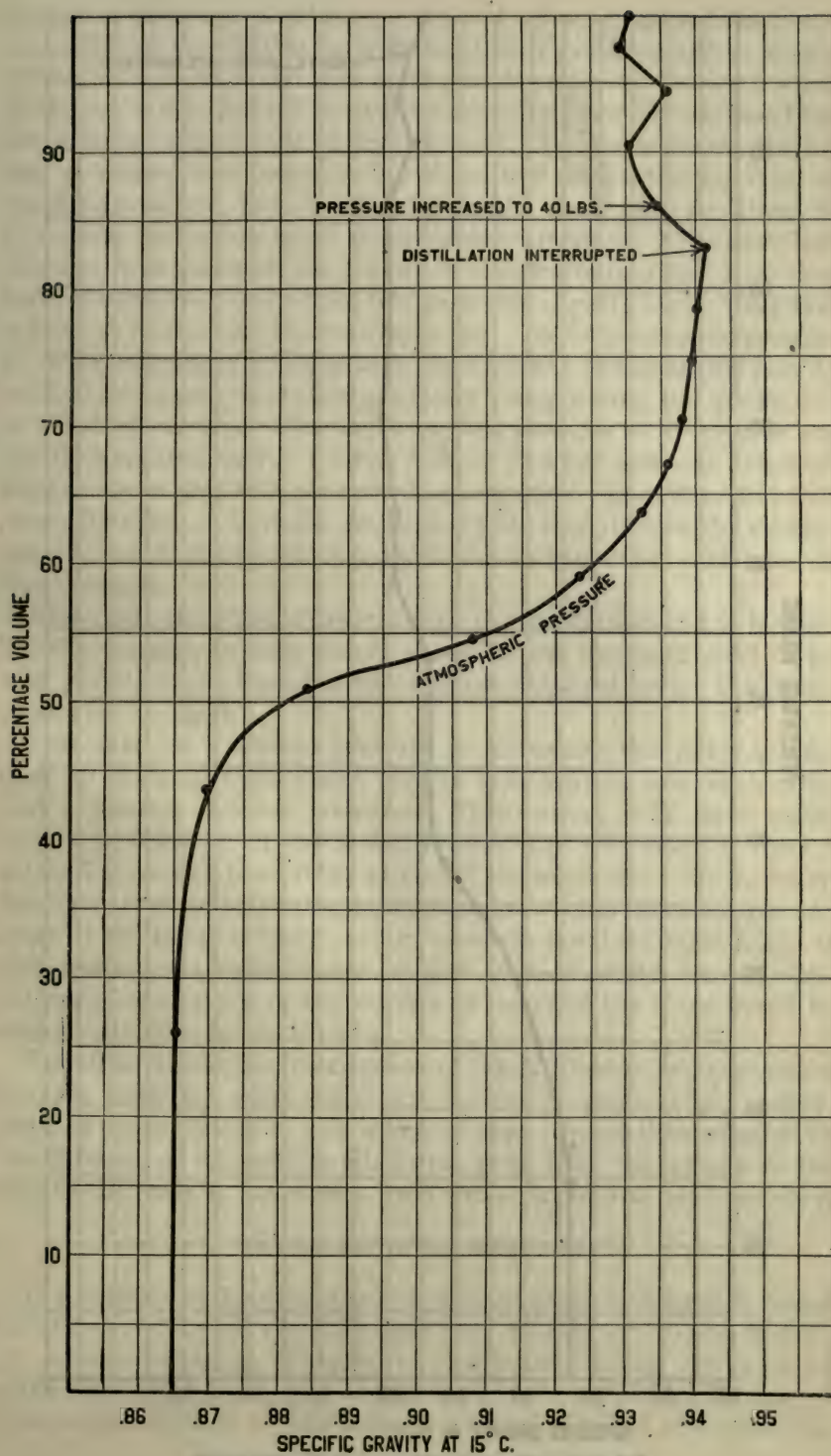


FIG. 5.—Curve showing fractionation of oil during run 16.

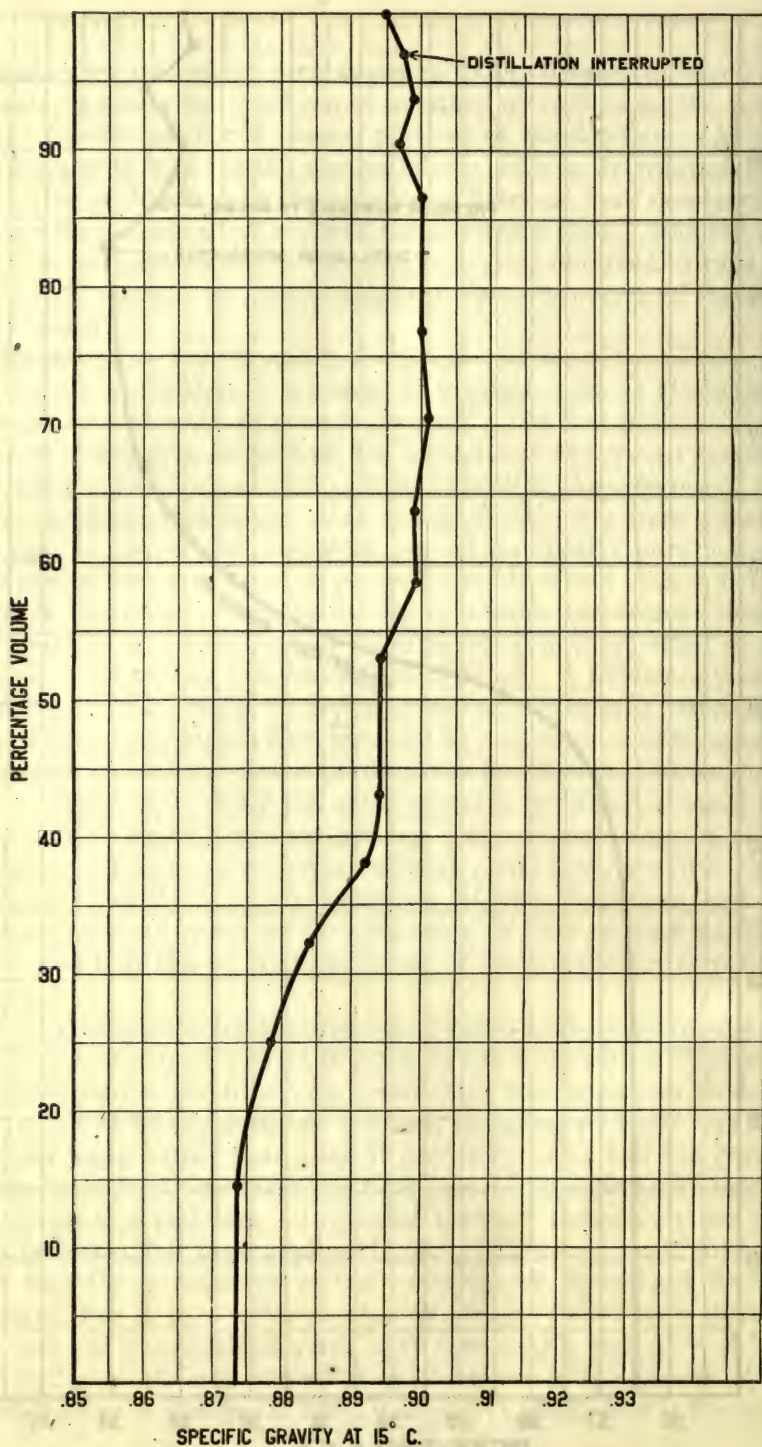


FIG. 6.—Curve showing fractionation of oil during run 20.

figure 7. This represents the gravities of different parts of the oil obtained during the distillation of chips $1\text{ inch} \times \frac{1}{2}\text{ inch} \times \frac{1}{8}\text{ inch}$ at atmospheric, at 20 pounds, and then at 40 pounds pressure. During the first of the run at atmospheric pressure the gravity gradually increased but never quite reached that of pure pine oil. By a continuous distillation at atmospheric pressure only about 50.6 per cent of the total oil could be removed, but on interrupting the distillation for about 14 hours and continuing again at atmospheric pressure, 7 per cent more of the oil was obtained, the gravity of the first part of this 7 per cent being much lower, and of the last part only slightly lower, than that of the last fraction of the continuous run. On increasing the pressure to 20 pounds about 16.8 per cent more oil was obtained, the gravity suddenly dropping and then gradually rising during the distillation of this 16.8 per cent. On increasing the pressure to 40 pounds and distilling continuously, a further yield of 19.5 per cent was obtained, the gravity of this 19.5 per cent dropping suddenly at first and then gradually rising. A similar additional yield was obtained by another interruption of the distillation, after which about 6 per cent more oil was obtained.

Here again the effect of interrupting the distillation and of increasing the pressure is very plainly shown in the increased yield of oil with gravity lower than the last fraction obtained before the conditions were changed.

This effect of increased pressure in increasing the yields is due, then, to bringing more steam and oil into contact with each other than is possible at lower pressures. This contact could result either from a penetration of the steam further into the wood or from a better flow of resin toward the surface of the wood, due to its increased fluidity. It is probable that both these have some influence, but the effect of the latter is quite certain, since, in the distillation made at high pressures, a considerable amount of rosin would collect in the bottom of the retort, or the outside of many of the chips would be coated with thin layers of rosin.

The effect due to the interruption of the distillation and continuing it again under the same conditions can not be explained so readily, but it is probably due to a slow flow of resin toward the surface or to the diffusion of the volatile oils in the resin from the interior of the chip to the resin at the surface from which the oil has been removed.

APPLICATION OF RESULTS.

In this study of the distillation of resinous wood by saturated steam the effects of different variables have been considered: (1) Size of chip, (2) pressure of steam, (3) speed of distillation, and (4) end point at which distillation is stopped, on (a) the yield of total oil, (b) the composition of the oil, and (c) the amount of steam required to remove the

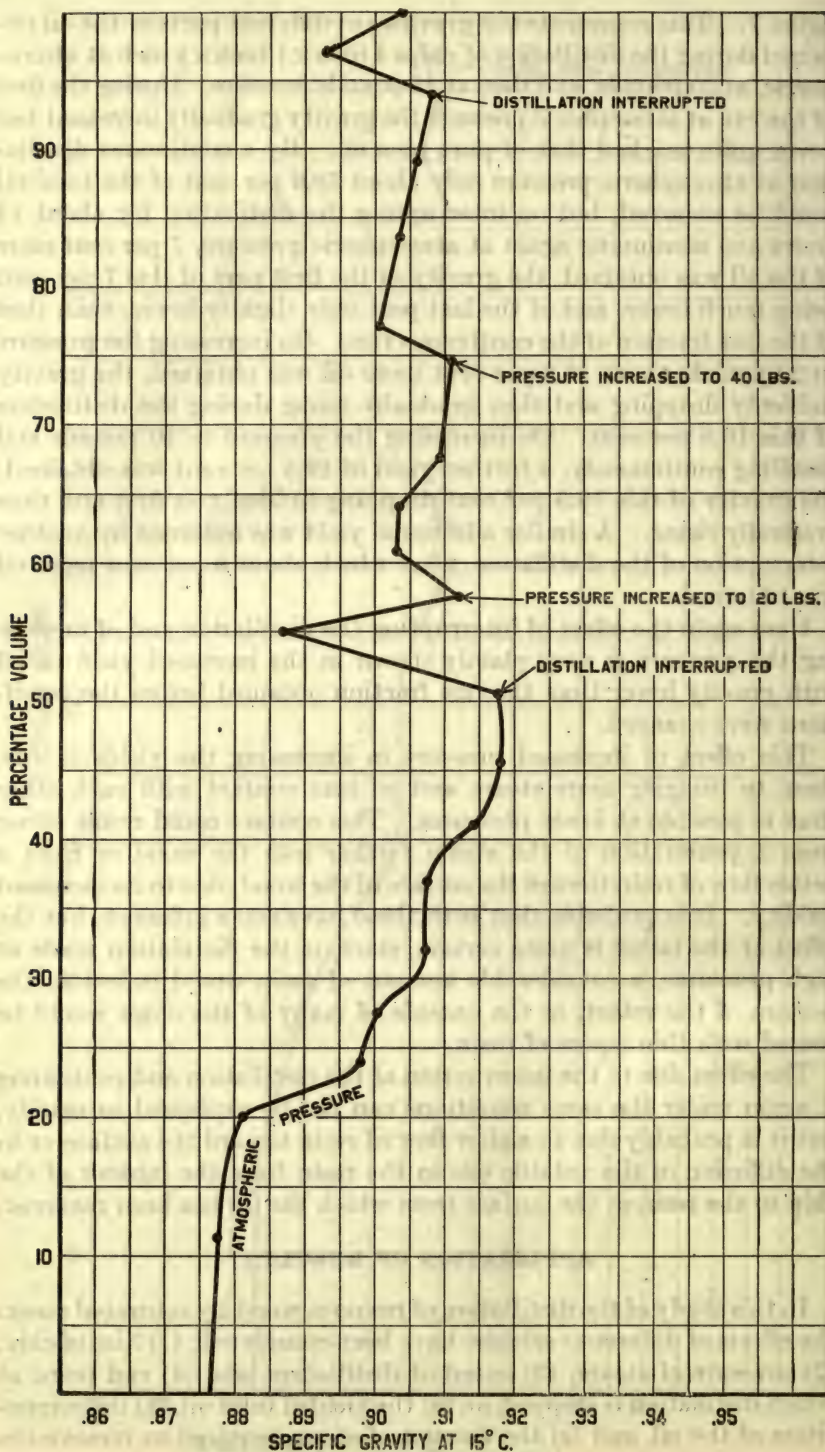


FIG. 7.—Curve showing fractionation of oil during run 7.

oil. It can be seen that there should be a certain combination of values for these variables which would give the most economical method of operation for a steam distillation plant; but there are other factors which must be taken into consideration in determining the proper combination of values. For example, the best size of the chip will not be determined entirely by the effect of size on yield and efficiency, but also by the relative costs of preparing different sized chips and the use to which the chips are to be put after steaming; the best pressure of steam will not be determined entirely by the effect of pressure on yield and efficiency, but also by the relative costs of high and low pressure steam and of apparatus for various pressures; the best speed for the distillation will not depend entirely upon the effect of speed on the yield of products and on the amount of steam required, but also upon the cost of steam and the overhead charges; the best end point at which to stop the distillation will not depend entirely upon the effect of end point on yield and efficiency, but also upon the cost of the raw material and the value of the products.

With the experimental data given, however, and with a knowledge of the various cost factors, which would naturally differ widely in different plants, it will be possible to decide readily on the most economical methods of operating.

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THE BALANCE, THE OVEN, AND THE UPPER PORTION OF THE CALORIMETER.

Issued September 20, 1912.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE—BULLETIN 110.

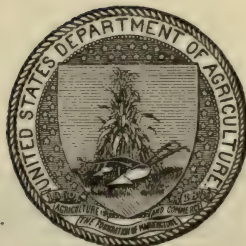
HENRY S. GRAVES, Forester

FOREST PRODUCTS LABORATORY SERIES.

THE SPECIFIC HEAT OF WOOD.

BY

FREDERICK DUNLAP,
Forest Assistant, Forest Service.



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HENRY S. GRAVES, *Forester.*

ALBERT F. POTTER, *Associate Forester.*

HERBERT A. SMITH, *Editor.*

BRANCH OF PRODUCTS.

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OLLISON CRAIG, *Assistant Engineer in Forest Products.*

ELOISE GERRY, *Microscopist.*

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LETTER OF TRANSMITTAL.

UNITED STATES DEPARTMENT OF AGRICULTURE,
FOREST SERVICE,
Washington, D. C., March 21, 1912.

SIR: I have the honor to transmit herewith a manuscript entitled "The Specific Heat of Wood," by Frederick Dunlap, Forest Assistant, and to recommend its publication as Bulletin 110 of the Forest Service.

Respectfully,

HENRY S. GRAVES,
Forester.

Hon. JAMES WILSON,
Secretary of Agriculture.

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THE SPECIFIC HEAT OF WOOD.

IMPORTANCE OF DETERMINING THE SPECIFIC HEAT OF WOOD.

The specific heat of wood—that is, the heat capacity of a given weight of wood compared with the heat capacity of the same weight of water—is one of the fundamental physical properties which indicate its intrinsic nature and ultimate structure. The need for reliable knowledge of this property was first revealed by a study of dry-kiln operations. Different operators use different methods in drying the same material for the same purpose; and, although their methods differ only in detail, their conceptions of the manner in which drying takes place differ enormously. In spite of the high grade of intelligence and engineering skill devoted to the operation of the larger kilns, the results are in no case entirely satisfactory. The cause is the lack of familiarity with the minute structure and physical properties of wood. In fact, there are no reliable values for those physical properties which control the transfer of heat¹ or water through wood. Knowledge of the specific heat of wood is necessary, however, not only in dry-kiln operation, but also in solving certain problems of the preservative impregnation of wood, the distillation of wood, and of all processes in which wood is subjected to a change of temperature. Though this study aimed solely to secure reliable values for specific heat,¹ it is hoped that it may also direct attention to the study of the other fundamental physical properties, so that eventually a basis may be secured for the formulation of a rational theory of the nature and ultimate structure of wood and of its action under different treatments. While the results are of practical service in commercial practice, they are even more useful in laboratory experiments.

APPARATUS AND METHODS.

The specific heat was determined by measuring in a calorimeter the quantity of heat given off by small cylinders of oven-dry wood in falling from a temperature slightly above that of boiling water to that of melting ice. The result in calories, divided by the difference

¹ Prof. William Kent, in "The Mechanical Engineer's Pocketbook" (8th edition, 1910, p. 536), gives the specific heats of four species of wood, the values ranging from 0.467 to 0.650. Prof. Kent does not cite the source of these figures, but they appear to rest on determinations made by J. R. Mayer, of Heilbronn, a contemporary of Joule. Cf. Péclet, E. *Traité de la Chaleur*, 4th edition, 1878, Vol. I, p. 606.

between the initial and final temperatures of the wood in degrees centigrade, and by its weight in grams, gives the mean specific heat of the wood through the range of temperature involved.

THE OVEN.

A copper drying oven, liquid jacketed, was used to bring the wood to the oven-dry condition and also to warm it before its introduction into the calorimeter. The oven was heated by gas. The jacket was filled with a mixture of water and glycerine, in about the proportion of 3 to 5, by volume. This mixture, when boiling, gave a temperature inside the oven from 5° to 10° above the boiling point of water. A reflux condenser maintained the constancy of the mixture in the jacket. The oven is shown in Plate I (frontispiece).

The temperature inside the oven was determined by means of an ordinary mercury-in-glass thermometer inserted through the top of the oven, with its bulb free in the air, near the center of the oven. This thermometer was calibrated in the oven against a carefully calibrated thermometer of the same type, and was read to twentieths of a degree centigrade.

THE CALORIMETER.

Bunsen's ice calorimeter,¹ in a slightly modified form, was used for measuring heat. The chief advantage of this calorimeter is that no radiation correction is necessary, because the introduction of heat is not indicated by a rise in temperature. This feature is particularly advantageous, since wood cools slowly.

The ice calorimeter depends for its indications upon the difference between the specific volumes of ice and water at the freezing point. It consists essentially of a mercury-sealed reservoir of water. After the water has been partially frozen the calorimeter is packed in melting ice, which prevents either freezing or thawing. The introduction of a hot body supplies heat and melts a portion of the ice, so that the amount of ice in the reservoir is diminished and the amount of water is increased while both remain at the melting point of ice. The heat produces no rise in temperature, but is entirely consumed in melting a portion of the ice. Since ice occupies 9 per cent more volume than does the water from which it is frozen, the melting of a portion of the ice in the reservoir causes the contents to contract. The measurement of this contraction indicates the amount of heat introduced.

The calorimeter used is shown in section in figure 1. The long glass tube at the axis is provided for the reception of the heated wood; its lower end, A, projects into the cylindrical jacket B. The upper end of this jacket is sealed to the tube, and the lower end opens into

¹ Bunsen, R. *Calorimetrische Untersuchungen*. Pogg. Ann. CXLI, pp. 1-31, 1870.

a narrow tube, C, which drops, curves upon itself, and rises past the side of the jacket to near the top of the central tube. The upper end of the tube C is enlarged into the cup D. These three parts, A, B, and C (with D), are the essential constituents of Bunsen's calorimeter. The body (B) of the instrument is filled with water above and mercury below, the mercury filling also the arm at the side and rising into the cup at the end of the arm. (See fig. 1.)

After partially freezing the water Bunsen packed this instrument in clean snow and attached a capillary glass tube to the top of C through a cork stopper. Then he noted, by the recession of the mercury meniscus in the capillary tube, the change in volume produced by the introduction of heat into the tube A. Bunsen's instrument was much smaller than the one used in these experiments. With it he determined the specific heats of calcium, indium, ruthenium, and zinc, using about one gram of material in each case.

Two modifications have been used in the present instrument. The first is the substitution of the reading arm, E (see fig. 1), for the capillary tube, which showed directly the contraction of the contents of the calorimeter. This reading arm¹ consists of an extension of the arm C by which it is

possible to establish and interrupt connection between the mercury in the calorimeter and that in a vessel, F, suitable for weighing. The vessel of mercury is weighed, and then connection between the mercury in the vessel and that in the calorimeter is established

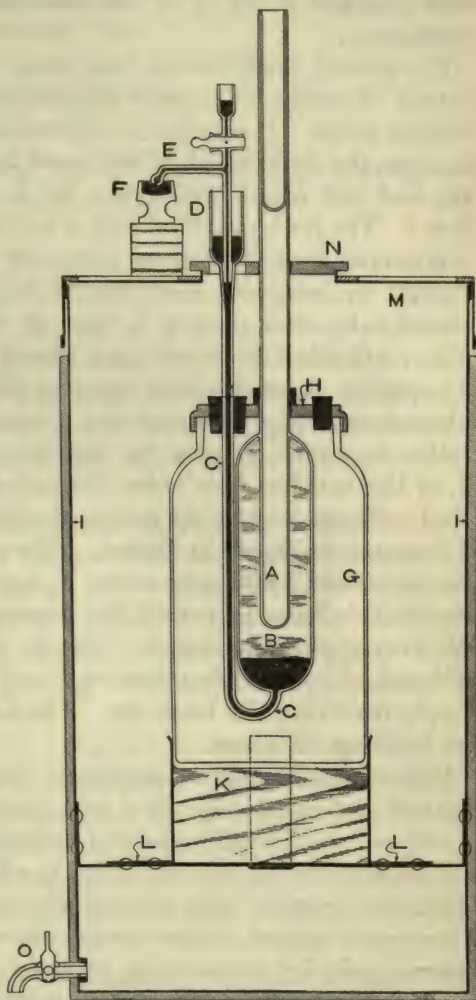


FIG. 1.—The ice calorimeter and ice jacket in vertical section.

¹ First described by Schuller and Wartha in 1877. Schuller, A., and Wartha, V. *Calorimetrische Untersuchungen*. Wied. Ann. II, pp. 359-383. 1877.

through the small opening in the tip of the arm E. After a certain interval of time this cup is removed and weighed again. The loss of weight tells the amount of mercury which the melting of the ice caused to be drawn into the calorimeter from the cup. In these determinations two cups were used, one of which was attached to the instrument while the other was being weighed. The cups were changed every 5 or 10 minutes during the course of each experiment.

The second modification was necessitated by the use of lake ice instead of newly fallen snow for maintaining the calorimeter at the freezing point. It consists in suspending the calorimeter in an empty glass jar, the air in which affords good insulation between the calorimeter and the ice in which the jar is packed. The idea is due to Boys.¹ The jar is provided with a vulcanite cover, H, which has holes to accommodate the central tube and the side arm. Suspension is effected by wrapping electrician's friction tape tightly around the central tube after passing it through this cover. A rubber stopper with a suitable hole is split and placed around the side arm to close the opening through which the arm passes. The jar is placed in an asbestos-covered galvanized-iron ice can, I, upon a block of wood, K, resting in a rack, L, near the bottom of the can. The metal cover, M, of the can has four holes through which the ice may be replenished. These may be opened or closed by revolving a circular piece of linoleum as shown in Plate I. The metal cover is also cut away in the center and a vulcanite cover, N, fitted into the opening. Cover N has suitable holes to permit the passage of the central tube and the side arm of the calorimeter. The ice in the can may, if desired, be replenished by removing the arm E and the metal cap M instead of by simply revolving the linoleum. The can is provided with a cock, O, for draining off water.

Before starting the experiments the calorimeter was thoroughly cleaned and cautiously filled with carefully prepared water of high purity and with freshly distilled mercury. To start the formation of ice, the calorimeter was immersed in a freezing mixture. In this way frondlike crystals were formed through the whole body of water. These were melted almost completely away and the water was then frozen again by evaporating ether in the central tube. The ice so formed was vitreous, developing a tubercular structure on thawing. Ether was used to renew the ice in the calorimeter from time to time.

The flow of mercury between the calorimeter and the weighed cup was noted for a sufficient period before and after each run to furnish a correction for heat lost or gained from sources other than the heated wood.

¹ Boys, C. V. On an addition to Bunsen's ice calorimeter. Phil. Mag. 5 ser. XXIV, pp. 214-217. 1887.

INTERPRETING THE INDICATIONS.

In order to interpret the changes in weight observed, it is necessary to know the weight of mercury drawn in the calorimeter when one calorie of heat is introduced. This amount can be calculated directly from physical data, and can also be determined experimentally. The method of calculation from physical data was pointed out by Bunsen, and is extremely simple. Let W be the weight in grams of mercury drawn in for one calorie: then

$$W = \left(\frac{V_i - V_w}{H} \right) D_m;$$

where V_i is the specific volume of ice, V_w is the specific volume of water, D_m is the density of mercury, and H is the latent heat of fusion of ice, all at 0°C .

But, since neither V_i nor H is known with sufficient accuracy, this calculation fails to give reliable results. Bunsen¹ actually calibrated his calorimeter by introducing a definite weight of water at 100°C . and noting the change which resulted. He then determined V_i in a separate experiment, and used the formula to calculate H . He found that the introduction of one mean calorie caused a contraction of 0.001133 cubic centimeter, which is the volume occupied by 15.41 milligrams of mercury.

Schuller and Wartha² repeated this calibration, using the same method: They found, as the mean of five very concordant results, that the introduction of one mean calorie caused the inflow of 15.442 milligrams of mercury. An accuracy greater than one-tenth of 1 per cent is claimed for this value, and it is generally recognized as the most reliable that has been secured. Velten³ found 15.47 for this value.

Dieterici⁴ accepted the value obtained by Schuller and Wartha, after discarding the last place as insignificant, and pointed out that it was the mean of the values obtained by Bunsen and by Velten. He accepted and used this result without checking it.

Von Than,⁵ Nessen,⁶ and Chappuis⁷ contributed nothing to the knowledge of this factor. Mond, Ramsay, and Shields⁸ followed

¹ Bunsen, R.; l. c.

² Schuller, A., and Wartha, V. *Calorimetrische Untersuchungen*. Wied. Ann. II, pp. 359-393. 1877.

³ Velten, A. W. *Das spezifische Wärme des Wassers*. Wied. Ann. XXI, 31-64. 1884.

⁴ Dieterici, C. *Ueber eine Bestimmung des mechanischen Aequivalentes der Wärme und über die spezifische Wärme des Wassers*. Wied. Ann. XXXIII, pp. 417-444. 1888.

⁵ V. Than, C. *Die Verbrennungswärme des Knallgases in geschlossenen Gefässen*. X, Ber. d. deutschen chem. Gesellschaft, pp. 947, 952. 1877. *Thermoschemische Untersuchungen*. Wied. Ann. XIII, pp. 84-105. 1881.

⁶ Neesen, F. *Ueber die spezifische Wärme des Wassers*. Wied. Ann. XVIII, pp. 369-386. 1883.

⁷ Chappuis, J. *Sur les chaleurs latentes de vaporisation de quelques substances très volatiles*. Ann. de Chem. et Phys. 6 ser. XV, pp. 498-517. 1888.

⁸ Mond, L., Ramsay, W., and Shields, J. *Ueber die Okklusion von Sauerstoff und Wasserstoff durch Platinschwarz*. II. Zeitsch. für Phys. Chemie, XXV, pp. 657-685. 1898.

Dieterici, but checked the value by determining the specific heats of lead and zinc.

The factor 15.44 was used in the present work. This value was checked at the end of the work by determining the heat of neutralization of hydrochloric acid with sodium hydroxide. (See Appendix.)

THE BALANCE.

A Spoerhase analytical balance (see Plate I), sensitive to one-fiftieth of a milligram, and a set of first-quality Sartorius weights, were used in all determinations of weight, but fractions of a milligram were disregarded in the regular series of weighings.

In weighing the cups of mercury a counterpoise was used to avoid wear of the larger weights. This counterpoise was a cup half filled with mercury and covered with a plate of glass sealed on with Canada balsam. Inasmuch as differences in the weight of the cups were alone significant, the weight of this counterpoise was not determined.

WEIGHT, TEMPERATURE, AND VOLUME DETERMINATIONS.

The green or air-dry cylinders were first brought to the oven-dry condition by being placed in the oven at about 105° centigrade for about two days and then weighed at intervals until their weight became constant to within 1 milligram for eight hours. This constant weight was recorded* in each case as the oven-dry weight of the specimen. The cylinder, before being weighed, was cooled in a stoppered test tube and then transferred to a vessel of known weight in which it was placed on the balance pan.

The oven-dry cylinder was brought to an even temperature all through by being held in the oven for at least 18 hours after the oven-dry condition had been reached, during the last half hour of which the temperature of the oven was kept constant to within one-tenth of a degree centigrade. The bulb of the thermometer was within a centimeter of the cylinder to be used in the ensuing run.

The transfer from the oven to the calorimeter was made in a pair of silvered Dewar tubes. The cylinder was placed in the smaller tube, fitting it loosely, and the smaller tube inserted, mouth first, into the larger one. These tubes were kept in the oven except when in use and always had the same temperature as the wood they carried.

At the end of the experiments the densities of all cylinders were found by measuring their volumes through the displacement of mercury in a graduated cylinder. This method failed only with red oak, into the vessels of which the mercury entered.

SELECTION AND PREPARATION OF THE WOOD.

The wood was taken from material on hand at the Forest Products Laboratory, and was selected to reveal variations in the specific heat of oven-dry wood due to three causes: (a) Position of the wood in the bole of the tree; (b) locality and site where grown; (c) species. The native species were botanically identified; of the four foreign species, two came with the names given. The species are as follows:

White pine.....	<i>Pinus strobus</i> L.
Longleaf pine.....	<i>Pinus palustris</i> Mill.
Red spruce.....	<i>Picea rubens</i> Sargent.
Western hemlock.....	<i>Tsuga heterophylla</i> (Raf.) Sargent.
Douglas fir.....	<i>Pseudotsuga taxifolia</i> (Lam.) Britt.
Red cedar.....	<i>Juniperus virginiana</i> L.
Mockernut hickory.....	<i>Hicoria alba</i> (L.) Britt.
Quaking aspen.....	<i>Populus tremuloides</i> Michx.
Beech.....	<i>Fagus atropunicea</i> (Marsh.) Sudworth.
Chestnut.....	<i>Castanea dentata</i> (Marsh.) Borkh.
White oak.....	<i>Quercus alba</i> L.
Red oak.....	<i>Quercus rubra</i> L.
White elm.....	<i>Ulmus americana</i> L.
Sugar maple.....	<i>Acer saccharum</i> Marsh.
Black gum.....	<i>Nyssa sylvatica</i> Marsh.
White ash.....	<i>Fraxinus americana</i> L.
Unknown wood, foreign, very light.	
Mai champah from Siam.	<i>Michelia</i> .
Unknown wood, foreign, heavier than water.	
Cocobola negra from Costa Rica.	<i>Lecythis costaricensis</i> Pittier.

The 16 indigenous species were chosen to include various forms of nonporous, ring-porous, and diffuse-porous woods. Their density varied from that of white pine to that of hickory. The four foreign woods were selected on account of their high or low density.

The great majority of the native woods were taken from short logs from the bole of the tree, usually from somewhere near breasthigh. Cylinders were cut in such a way that their axes ran with the grain of the wood and were turned on a lathe. Each cylinder was about 17 millimeters in diameter and 3 to 9 centimeters long, the length depending upon the weight, which was preferably about 6 grams. Each cylinder was marked with an Arabic numeral to indicate the species and the log from which it was taken and also with a capital letter to distinguish it from other cylinders cut from the same piece.

To learn the variation due to the position of the wood within the bole of the tree and to the locality and site where the wood was grown a single species was selected. Douglas fir was chosen because of its wide range both geographically and ecologically and because of the great difference in quality shown by wood of this species from different localities. Sticks collected by members of the Forest

Service in three different parts of its range and two sticks from different sources purchased in Madison, Wis., were used. The material showed a variation of from 3 to 20 rings per centimeter and a difference in density of from 0.40 to 0.59. A piece was cut from just above the stump and another from the bole near the crown, and from each of these pieces cylinders were made representing successive positions between the pith and the bark. No branch wood was studied. When the many determinations on Douglas fir showed no consistent variation of specific heat due either to the position of the wood in the bole or to the place where the tree grew it seemed fairly certain that no such variation existed in any species. Nevertheless, three sticks each of red oak and sugar maple were secured from widely separated localities and possibly from different though closely related species. For these, likewise, no difference in specific heat could be detected within the species. Therefore, when the study of other woods was taken up no attention was paid to the part of the tree from which the cylinders were taken or to the locality whence the wood came.

RESULTS.

The chief results of this work are two: First, the mean specific heats of 20 species of wood have been accurately measured over the interval between 106° C. and 0° C.; second, preliminary results have been secured which indicate a great variation of specific heat with temperature. Also an increase of specific heat was encountered which was apparently due to steaming.

MEAN SPECIFIC HEAT.

A summary of the determinations of the mean specific heat for each species is given in Table 1, in which the values corrected by the frequency curve (see appendix) are shown in the last column. Results (calculated) for each cylinder of wood are given in Table 5, appendix.

TABLE 1.—*Specific heat of oven-dry wood—averages by species.*

Species.	Number of determinations. ¹	Density.	Mean specific heat 100°-0° C.	
			Calculated from observations.	Corrected. ²
Douglas fir.....	37	0.48	0.325	0.327
Longleaf pine.....	2	.68	.335	.337
White pine.....	2	.25	.329	.331
Western hemlock.....	2	.45	.320	.322
Red spruce.....	2	.39	.330	.332
Red cedar.....	2	.46	.322	.324
Red oak.....	13	.62	.329	.331
White oak.....	3	.78	.323	.325
Chestnut.....	4	.32	.315	.317
White elm.....	2	.64	.323	.325
Mockernut hickory.....	2	.84	.325	.327
White ash.....	3	.63	.325	.327
Sugar maple.....	12	.66	.325	.327
Quaking aspen.....	2	.42	.327	.329
Beech.....	2	.75	.324	.326
Black gum.....	2	.52	.323	.325
Cocobola negra from Costa Rica.....	2	.92	.325	.327
Mai champah from Siam.....	2	.32	.321	.323
First unknown wood.....	2	1.10	.322	.324
Second unknown wood.....	2	.23	.320	.322

¹ For the rejection of 16 determinations see appendix.² The determination of the correction is discussed in the appendix.

The average of the calculated values for the specific heat of wood obtained by giving equal weight to each of the 20 species studied is 0.3244; that obtained by giving equal weight to each of the 100 runs shown in Table 1 is 0.3249. The close agreement between these averages, in spite of the fact that one-half of the runs was made upon Douglas fir and maple, shows strikingly how little influence species has upon the specific heat of wood. The average referred to hereafter, unless otherwise specified, will be that giving equal height to each of the 100 runs 0.325 calculated, or 0.327 corrected.

The table shows the specific heat of longleaf pine to be about 3 per cent greater than the average, and that of chestnut to be the same amount less. But the values for these species are the mean of only a few results, and are therefore much less reliable than the average with which they are compared. A consideration of the probable error of the observations indicates that this difference must be at least 4 per cent to acquire significance. The differences shown in the table may be real, but are too small to be significant, and furnish no basis for drawing a distinction between the specific heat of different species or of conifers and broadleaf trees. Therefore, the mean specific heat of oven-dry wood may be considered 0.327, and this figure remains nearly constant for all wood.

EFFECT OF LOCALITY.

The variation in specific heat within a species due to the locality in which it is grown was so small and inconsistent as to furnish no basis for concluding that locality influences the specific heat of

wood. The values obtained by the experiments along this line are given in Table 2:

TABLE 2.—*Results of determinations on the same species of wood grown in different localities.*

Species.	Source.	Stick.	Mean specific heat 106°-0° C.	Number of determinations.
Douglas fir.....	Commercial.....	5	0.3248	9
	Portland, Oreg.....	6	.3286	2
	Commercial.....	7	.3288	2
	Denver, Colo.....	8	.3232	5
	Snoqualmie National Forest.....	9	.3282	4
Sugar maple.....	Glandon, Wis.....	10	.3277	12
	do.....	11	.3242	4
	do.....	12	.3264	4
Red oak.....	Corbin Park, N. H.....	14	.3250	4
	Richland Parish, La.....	15	.3320	4
	Glandon, Wis.....	18	.3298	4
	Brandywine, Md.....	19	.3286	4

HEARTWOOD AND SAPWOOD.

The differences met in cylinders taken from the heartwood and sapwood on a line passing from the pith of a tree to the bark are even smaller than the differences in a species grown in different localities. The results are given in Table 3:

TABLE 3.—*Results of determinations on heartwood and sapwood of Douglas fir.*

Material.	Cylinder.	Mean specific heat 106°-0° C.
Heartwood.....	10D	0.3258
	10F	.3260
	10H	.3260
Sapwood.....	10J	.3259
	10L	.3262

CORRECTED RESULTS.

The above results were generalized by means of a frequency curve. (Appendix, p. 21.) This curve revealed the fact that the results are subject to a systematic error which tends to make the average result of 0.325 too low by about five-tenths of 1 per cent. This error, there is no reason to doubt, has its origin in the loss of heat during the transfer of the specimen from the oven to the calorimeter. Applying this correction gives a corrected value of 0.327, which is the most acceptable result for the mean specific heat of oven-dry wood through the interval between 106° C. and 0° C., and is believed to be correct within six-tenths of 1 per cent.

The two following rules may be laid down for the practical use of these results:

1. When the particular species, the specific heat of which is desired, is one of the eighteen named in Table 1, use the corrected value given in the last column of the table.

2. When the particular species is not given in Table 1, or is unknown, or when the wood of more than one species is lumped together in unknown proportions, use the value 0.327.

VARIATION IN SPECIFIC HEAT WITH TEMPERATURE.

In determining the variation in specific heat with temperature, the mean specific heat between 106° C. and 0° C. was taken as one point. Runs were made from approximately 65° and 25° to determine the mean specific heats between each of these temperatures and 0° C. A larger number of intervals will be necessary before the shape of a curve showing the variation in specific heat with temperature is known. The present results can be considered as only preliminary because the peculiar precautions taken to keep the wood in an oven-dry condition at these temperatures introduced errors which were not entirely removed by correction. The results are given in Table 4.

TABLE 4.—*Results of determinations with different initial temperatures.*

Run.	Cylinder.	Initial temperature.	Mean specific heat.
75	14C	109.40	0.314
76	19D	109.05	.326
124	18B	23.20	.280
125	19D	23.20	.280
126	14C	23.10	.281
132	14C	62.95	.299
133	18B	63.85	.286
134	19D	63.35	.286
136	22A	68.05	.297
137	25B	68.45	.295
139	18B	67.00	.289
140	19D	66.50	.286

The highest values obtained for each temperature are the most probable. Even these indicate an unusually large variation of specific heat with temperature.

By plotting the values for the two lower temperatures shown in the table and that for 106° C. already obtained, it was found that the specific heat of wood falls to the neighborhood of 0.266 at 0° C. Therefore, when represented by a straight-line formula, the value of the true specific heat at temperature t is indicated by the following equation:

$$\text{Specific heat} = 0.266 + 0.00116t$$

The mean specific heat through any interval of temperature is, under this assumption, of course equal to the true specific heat at the mean temperature. The values given are subject to correction when a large number of runs from different temperatures have been made. They are advanced at the present time merely to set forth the best knowledge at hand.

EFFECT OF STEAMING ON SPECIFIC HEAT.

The wood used in five runs, Nos. 47, 48, 49, 50, and 51, was placed in the oven at 110° C. while still moist, and dried in nearly saturated air at this temperature. Of these five pieces of wood three showed a greatly increased specific heat. Accordingly it appears possible that exposure to moisture at high temperature increases the specific heat of wood. If this is true, other properties, and with them technological characters, may also change. This behavior needs further study.

APPENDIX.

THE CALIBRATION OF THE CALORIMETER.

The value 15.44 milligrams for the weight of mercury drawn into the calorimeter by the introduction of one calorie of heat was checked by measuring the heat of reaction of hydrochloric acid and sodium hydroxide. The advantage of this method of calibration is that it avoids all measurements of temperature and all error due to loss of heat in dropping a hot body down the tube of the calorimeter.

Half-normal solutions were used. The acid was carefully prepared and standardized, both against solutions of known strength and by determining the chlorine as silver chloride. Ten cubic centimeters were used. The alkali was prepared with less care and used in excess, the use of the ice calorimeter requiring that but one reagent be present in known amount and high purity. The weight of mercury drawn in in three runs was 1.1731, 1.1729, and 1.1725 grams; the average being 1.1728 grams. For the solution used (0.5078 normal) this gave 14,958 for the heat of neutralization.

Such calibration is not entirely satisfactory because this value has been determined before only at higher temperatures and the specific heats of dilute solutions are not known with sufficient accuracy to permit the calculation from one temperature to another. Julius Thomsen¹ has studied the change of heat of neutralization with temperature and gives values for this reaction at 25° C. and 10° C. The value obtained appears high when compared with these two, but it is to be noted that the accepted value for this heat of reaction at 18° is low by the same comparison. In fact, the three values for 18°, 10°, and 0° C. lie nearly in a straight line. It is hoped that the heat of this reaction at zero will be accurately determined by other methods and so remove the only objection to the use of this excellent method of calibration.

PROCEDURE IN GENERALIZING RESULTS.

Upon completion of the measurements and calculation of the results it becomes necessary to determine the best representative value and to measure the consistency of the results, and, on the basis of this measure, to form an estimate of the reliability of the representative value determined. Accurate results are always discordant,

¹ Thermochemische Untersuchungen. Bd. I, p. 64.

due to chance errors which are unavoidable; in addition they may be biased by the presence of systematic errors. Chance errors obey certain mathematical laws which have been formulated by Laplace; their measure is obtained by purely mathematical processes and consists either of the mean error or the "probable" error. Systematic errors are susceptible of no mathematical treatment, and, unless they are very simple, their discussion leads to no numerical corrections. Their presence is best revealed by the form of the frequency curve and its relation to the arithmetic mean.

REJECTED RESULTS.

In Table 5 are displayed the material used in each of the 116 runs made from near 106°C. , the number and date of each run, the corrected observations, and the calculated results. The records of each one of these runs were carefully scrutinized, and where there was valid reason for doubting the fitness of any result it was rejected. Rejections were made for two reasons:

1. In the preparation of some of the material the wood was exposed to temperatures of 110°C. while still wet and dried in nearly saturated air at this temperature. Such treatment darkened the color of the wood, produced abnormal shrinkage, and, in some cases, appeared to raise its specific heat. To avoid the possible effects of such drastic treatment the results from the five runs, Nos. 47, 48, 49, 50, and 51, for which such material was used, were rejected entirely and a notation to that effect entered in the column headed "Remarks."

2. While nearly all of the runs were made from an initial temperature between 102° and 112°C. , three runs, Nos. 23, 24, and 25, were made from temperatures above 120°C. The departures of these runs from the average of the remaining results is not sufficiently consistent to warrant their rejection; but inasmuch as there are indications that the specific heat of wood varies greatly with temperature, the results of these three runs are also rejected.

The arithmetical mean of the remaining results was now calculated as a first approximation to the best representative value, and the criterion of permissible discordance was applied to the individual results. One of the practical rules resulting from the laws governing the behavior of chance errors is, that if *one* result of a series differs from the average of the *other* results by an amount which is more than four times the average deviation of the other results, the discordant result should be rejected, because to include it lowers the reliability of the average. If, in the application of this rule, the doubtful result is included in the average, the permissible discordance is increased and the criterion made looser. Obviously any result whose rejection is indicated under this application would certainly be

rejected if the rule were followed strictly. The advantage of applying the rule in this way is that under it more than one result may be rejected at a time.

After the omission of the eight runs already rejected, 108 results are left for further consideration. Their average is 0.320, and their mean discordance ± 0.011 ; so that all results greater than 0.364 and less than 0.276 are properly rejected. This includes four runs, Nos. 4, 16, 22, and 26, and leaves 104 results to which the same criterion is again applied. The average is now raised to 0.324, and the average discordance reduced to 0.006, so that results greater than 0.348 and less than 0.300 are properly rejected. Again, the results of four runs, Nos. 2, 81, 93, and 100, are marked for rejection and an even hundred results remain. Their mean value is 0.32486, their mean discordance 0.00518, and only results above 0.345 and below 0.304 can be rejected. None falls outside these limits. It is noteworthy that every one of the eight results rejected in the two successive applications of this criterion fell below those retained. The reason why all the faulty results were low and none were high will be shown later in discussing the reliability of the average in the light of the frequency curve.

The 100 results finally indicated for retention are now searched for systematic variation demanding corrections. The most obvious correction to apply would be for the varying temperatures at which the wood entered the calorimeter. Accordingly the results are tested for variation with initial temperature. This is done graphically, as shown in figure 2, which is made by plotting the individual results, giving each an abscissa determined by its temperature and an ordinate determined by its specific heat. The result is to scatter the points over the paper with seemingly no well-marked trend. But the trend is more clearly revealed when the results for successive intervals of temperature, such as half degrees, are averaged, as shown in the figure. These averages vary considerably, their range extending from 0.321 to 0.330, but the course of the line joining them is generally horizontal. Should any correction be decided upon, it would be an inverse function of temperature, which is the opposite of what results at lower temperatures indicate and would be small. Therefore a correction for temperature appears unwarranted.

Possible variation with species, density, etc., have already been disproven.

FREQUENCY CURVE.

The next step is the construction of the frequency curve shown in figures 3 and 5; figure 3 shows the development of this curve. The results of the separate runs were first arranged in order of magnitude, beginning with the smallest, 0.308, and ending with the largest,

0.340. Between these extremes is a difference of 32 units in the third decimal. It is convenient, then, to group the results into 32 classes, those lying between 0.308 and 0.309 forming Class A; those between 0.309 and 0.310 forming Class B, etc. Convenience is the only reason for making 32 classes. Forty or 20 or 25 might as correctly have been formed. Fifty would have been too many, as there would be an average of but two results in each class. Five would have been too few, not because 20 results in each class are too many but because five points would not show all the properties of the curve. The number of results in each one of the 32 classes formed is shown by the ordinates of the successive points on the lowest of the broken

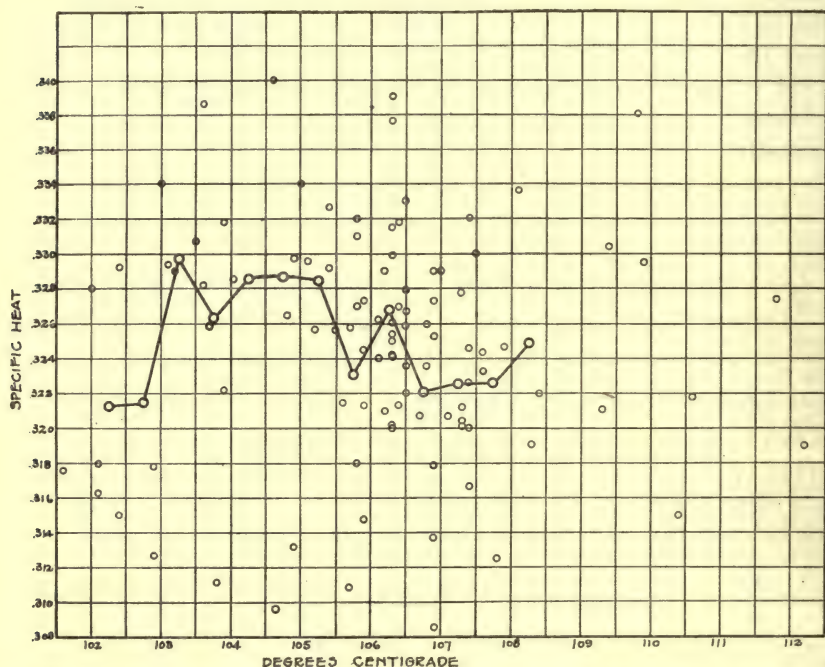


FIG. 2.—Distribution of results through range of initial temperatures, and average results for each half degree.

lines in figure 3. The sum of the ordinates under this line is 100. The variations in the successive ordinates indicate that the classes have been made too small. The pairing of successive classes AB, BC, CD, etc., and taking the number of observations in each pair, gives the second broken line in which, although the variation of successive ordinates has been reduced considerably, still further reduction appears advisable. For this purpose successive classes are united in threes, thus ABC, BCD, CDE, etc. When the number of results in each group of three is plotted, the successive frequencies show a much smaller irregular variation, as indicated in the upper broken

line in figure 3. By continuing to form successively larger groups smoother and smoother lines can be obtained, but it is better to make further adjustment in another way, leaving the work just performed with the decision to use groups of three successive classes in plotting the final curve.

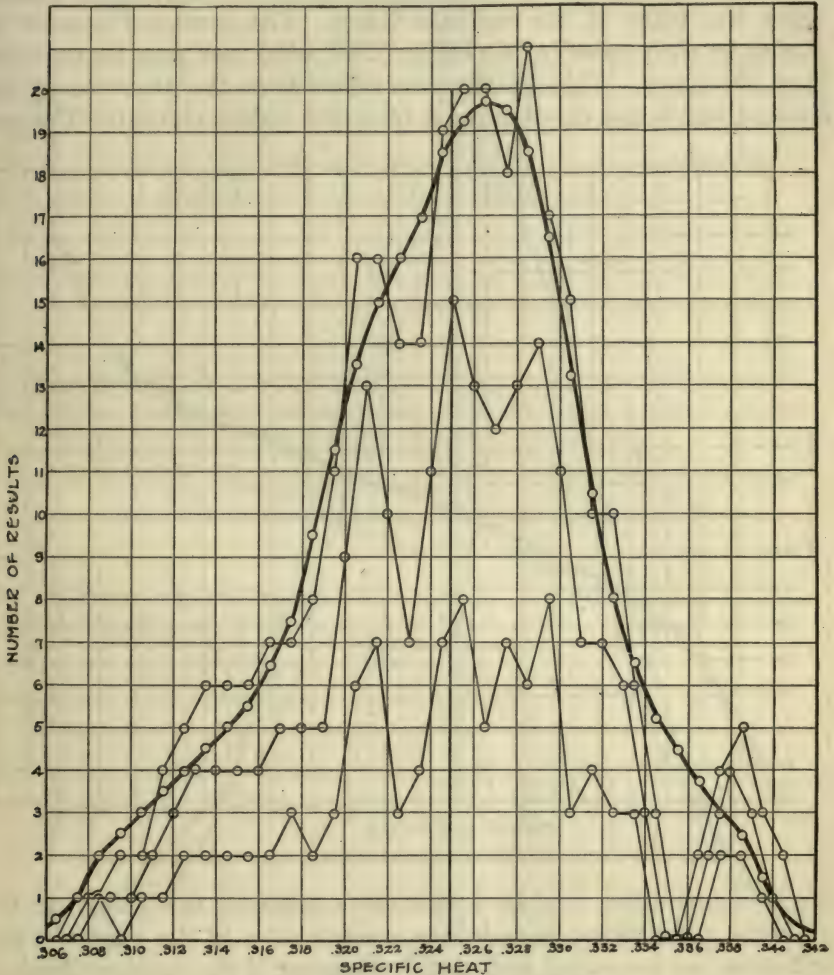


FIG. 3.—Frequency curve and its development.

The essential feature of the preceding method of grouping is the application of a correction to the number of results in each class according to the number of results in the two adjacent classes. Of course the graphical method of rounding off the results immediately suggests itself, and is used in the distribution curve, figure 4. The ordinates indicate specific heats and the abscissas the number of results. The individual results, arranged in order of their magnitude, are placed at equal distances apart along the horizontal axis; and each is

given the ordinate indicated by its magnitude. Connecting these in order gives an irregular broken line. But a regular curve, whose form approaches that of the cubical parabola ($a^2y=x^3$), can be so drawn as to pass through many of the individual results and very close to the remainder. The abscissa intercepted by this curve at any ordinate indicates the corrected number of results below the value of the ordinate taken. The corrected number of results in each class (ABC, BCD, CDE, etc.) can thus be obtained from the curve. This distribution differs from the observed by the amount which the curve departs from the points plotted. The cor-

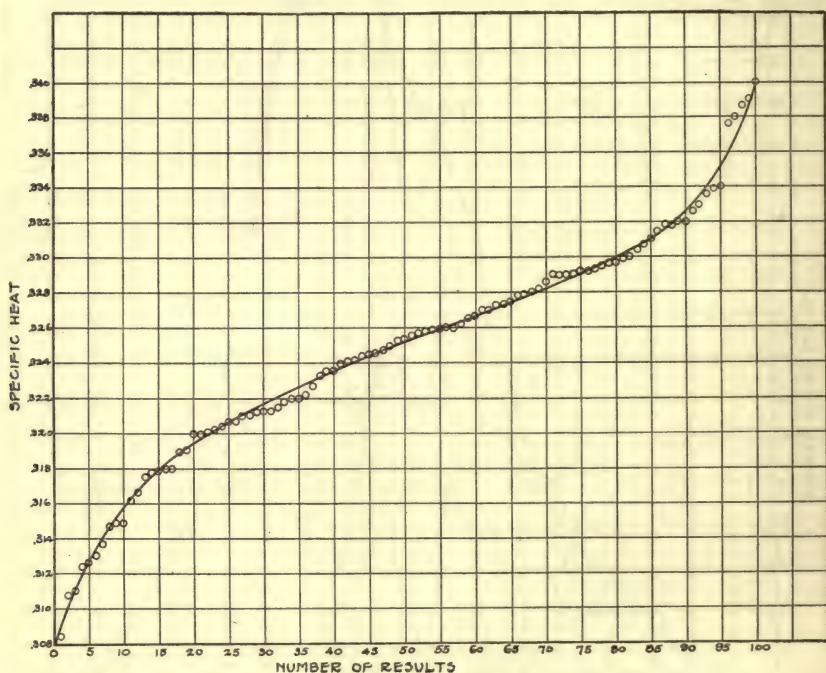


FIG. 4.—Curve for correcting distribution of results for a smooth frequency curve.

rected distribution, read from this curve, is nearer the truth than the observed distribution because the irregularities in the observed distribution are unnatural and would disappear if the number of points were greater.

The frequency curve shown in figures 3 and 5 is obtained from the corrected distribution of results derived from figure 4. It is entered on figure 3 merely to show its relation to the broken lines indicating the distribution of the results actually observed and is repeated on figure 5 for further discussion. This curve furnishes the best information which can be secured regarding the reliability of the mean value of the specific heat of wood.

This frequency curve is not a regular curve, though its grosser irregularities have been removed and its essential characteristics more clearly revealed. The irregularities left are best revealed by a comparison with the true frequency curve, $y=e-x^2$, shown in figure 5. The proportions of the true curve are obtained by giving it the same height and the same modulus as the curve under discussion.

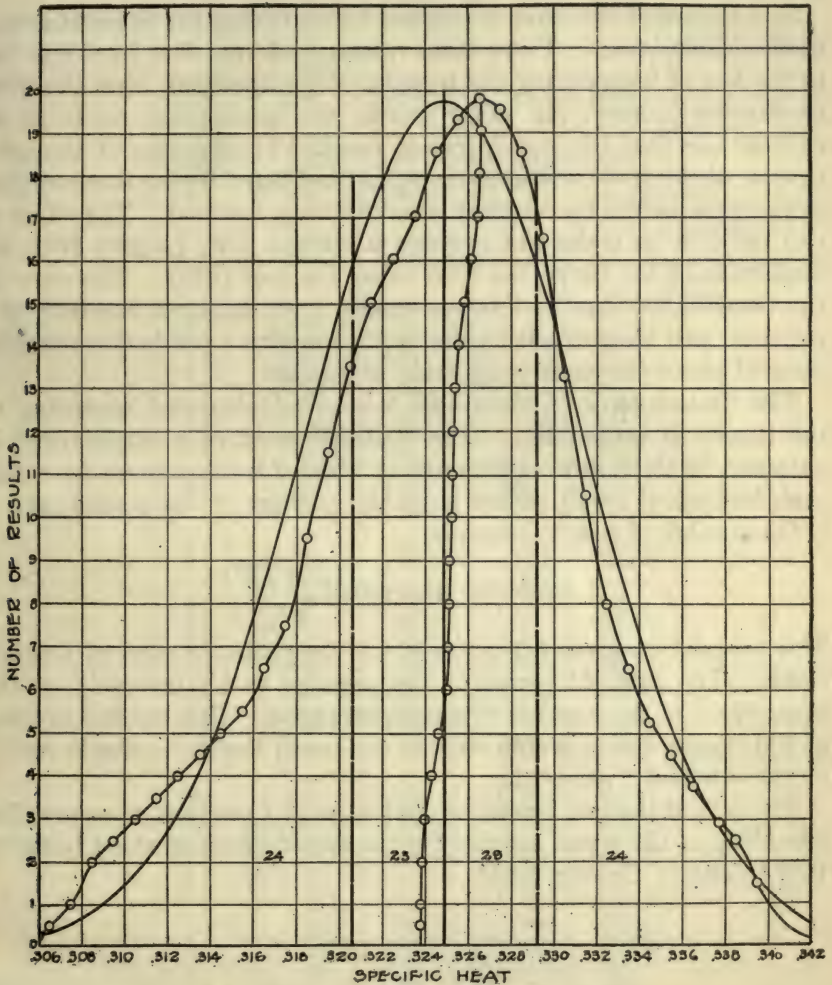


FIG. 5.—Empirical and theoretical frequency curves.

Its apex is placed on the ordinate of the average of the observed results. The comparison shows two ways in which the curves differ: (1) the empirical curve is too broad below; and (2) its crest is bent to the right toward the higher values of the specific heat. This inclination is clearly shown by the course of the median line of the empirical curve and its deviation from the median line of the true or

theoretical frequency curve which is formed by the ordinate over the average specific heat. The way in which the empirical curve differs from the true curve indicates the presence of a systematic error which tends to make the individual results too low by a varying amount. It was the exaggerated prominence of this error which led to the rejection of the results of eight runs under the procedure followed on page 20.

The nature of the error is learned by reviewing the apparatus and methods employed. There is no reason to doubt that its origin lies in the loss of heat during the transfer of the specimen from the oven to the calorimeter. In other words, the assumption made in all calculations that the hot specimen reached the interior of the calorimeter at the same temperature as that indicated by the thermometer in the oven before its removal is not entirely justified. The effect of this error is to make the average 0.325 too low; judging from an inspection of the curve the true value lies near 0.327. The error in the result 0.325 due to this systematic error is about one-half of 1 per cent, and the corrected value 0.327 contains a residual systematic error of about the same magnitude or smaller.

The chance error of the result is readily calculated according to the theory of probability. The probable error of a single result is obtained by the following formula, in which d is the amount by which each individual result differs from the average of the results, and n is the number of results averaged:

$$\text{probable error} = 0.67 \sqrt{\frac{\sum d^2}{n-1}}.$$

This calculation gives 1.3 per cent for the probable error of a single result. The probable error of the average varies inversely as the square root of the number of results averaged. Thus for the average of 100 results the probable error is one-tenth that for a single result, or one-tenth of 1 per cent.

The sum of the two errors, or six-tenths of 1 per cent, measures the reliability of the result secured for the specific heat of wood between 106° C. and 0° C.; viz, 0.327.

TABLE 5.—*Determinations of the specific heat of wood.*

Species.	Cylinder mark.	Run No.	Date.	Weight of mercury.	Weight of cylinder.	Temperature.	Specific heat.	Remarks.
Douglas fir.....	1 A	26	1911. Jan. 14	Mg. 4,781	Grams. 11.238	°C. 107.9	0.2552	Rejected in generalizing results. (See appendix.)
Do.....	1 A	27	Jan. 17	5,908	11.238	107.4	.3167	
Do.....	1 B	24	Jan. 13	6,928	10.988	123.9	.3293	Rejected in generalizing results; high initial temperature.
Do.....	1 B	15	1910. Dec. 22	5,486	10.988	103.8	.3112	
Do.....	2 A	2	Nov. 1	7,033	14.757	104.9	.2940	Rejected in generalizing results. (See appendix.)
Do.....	2 A	21	1911. Jan. 6	7,337	14.757	102.9	.3127	
Do.....	2 B	22	Jan. 7	3,072	12.349	102.0	.1580	Do.
Do.....	2 B	23	Jan. 13	7,800	12.349	120.2	.3404	Rejected in generalizing results; high initial temperature.
Do.....	2 B	16	1910. Dec. 23	4,733	12.349	103.4	.2401	Rejected in generalizing results. (See appendix.)
Western hemlock.....	3 A	3	Nov. 1	5,935	11.763	102.9	.3178	
Do.....	4 A	1	Oct. 31	5,000	9.510	105.9	.3213	
Douglas fir.....	5 A	4	Nov. 14	4,836	10.863	106.9	.2698	Do.
Do.....	5 A	14	Dec. 22	5,661	10.807	103.1	.3294	
Do.....	5 B	6	Dec. 7	5,442	10.312	103.5	.3307	
Do.....	5 C	7	Dec. 8	5,154	10.355	101.6	.3176	
Do.....	5 D	8	do.....	5,113	9.826	102.4	.3292	
Do.....	5 E	9	do.....	5,458	10.506	102.0	.3280	
Do.....	5 F	10	Dec. 9	5,000	10.030	102.1	.3163	
Do.....	5 G	11	Dec. 13	4,548	9.126	102.4	.3150	
Do.....	5 H	12	do.....	5,382	10.747	102.1	.3180	
Do.....	5 I	5	Dec. 1	4,679	8.757	105.1	.3296	
Do.....	5 I	13	Dec. 22	4,623	8.713	103.0	.3339	
Do.....	6 A	19	1911. Jan. 6	7,354	14.017	103.6	.3282	
Do.....	6 B	20	do.....	6,377	12.167	103.2	.3290	
Do.....	7 A	17	Jan. 3	5,111	9.793	103.7	.3259	
Do.....	7 B	18	Jan. 5	5,460	10.263	103.9	.3318	
Do.....	8 A	37	Jan. 24	6,359	11.744	106.3	.3299	
Do.....	8 B	38	do.....	6,861	12.628	107.0	.3290	
Do.....	8 E	30	Jan. 18	6,245	12.014	107.8	.3125	
Do.....	8 G	29	do.....	6,424	12.102	107.3	.3204	
Do.....	8 H	28	do.....	6,109	11.345	107.6	.3244	
Do.....	9 E	33	Jan. 20	6,079	11.040	107.4	.3320	
Do.....	9 F	32	Jan. 19	5,953	11.163	108.3	.3191	
Do.....	9 J	31	do.....	6,711	12.062	108.1	.3336	
Do.....	9 K	25	Jan. 13	6,328	10.807	124.0	.3059	Rejected in generalizing results; high initial temperature.
Do.....	10 A	34	Jan. 20	5,241	9.461	106.3	.3376	
Do.....	10 B	35	do.....	5,271	9.767	106.2	.3290	
Do.....	10 C	36	Jan. 21	5,385	9.822	104.6	.3400	
Do.....	10 D	103	Apr. 22	2,684	5.044	105.7	.3258	
Do.....	10 E	104	do.....	3,194	5.979	106.3	.3254	
Do.....	10 F	105	Apr. 24	3,080	5.760	106.3	.3260	
Do.....	10 G	106	do.....	3,388	6.346	106.8	.3236	
Do.....	10 H	107	do.....	3,654	6.795	106.8	.3260	
Do.....	10 I	108	Apr. 25	3,182	6.022	106.7	.3207	
Do.....	10 J	109	do.....	3,568	6.656	106.5	.3259	
Do.....	10 K	110	do.....	2,917	5.435	106.5	.3267	
Do.....	10 L	111	do.....	3,776	7.066	106.1	.3262	
Sugar maple.....	11 A	39	Mar. 8	6,285	11.415	110.6	.3218	
Do.....	11 B	40	do.....	6,332	11.322	109.9	.3295	
Do.....	11 C	41	Mar. 9	6,335	11.347	109.4	.3304	
Do.....	11 D	43	do.....	5,937	11.055	110.4	.3150	
Do.....	12 A	42	do.....	7,524	13.885	109.3	.3211	
Do.....	12 B	44	Mar. 10	7,005	12.220	109.8	.3380	
Do.....	12 C	45	Mar. 11	6,631	12.001	112.2	.3190	
Do.....	12 D	46	do.....	6,665	11.795	111.8	.3274	
Do.....	13 A	47	Mar. 17	7,298	12.972	104.5	.3486	Rejected in generalizing results. Steamed wood used.
Do.....	13 B	48	Mar. 18	7,216	13.000	104.4	.3441	Do.
Do.....	14 A	52	Mar. 24	6,748	12.469	107.9	.3247	
Do.....	14 B	53	do.....	7,202	13.269	107.3	.3278	
Do.....	14 C	54	do.....	6,541	12.154	107.4	.3246	
Do.....	14 D	55	do.....	6,719	12.561	107.4	.3227	

TABLE 5.—*Determinations of the specific heat of wood—Continued.*

Species.	Cylinder mark.	Run No.	Date.	Weight of mercury.	Weight of cylinder.	Temperature.	Specific heat.	Remarks.
			1911.	Mg.	Grams.	° C.		
Red oak	15 A	56	Mar. 27	5,986	11.061	103.6	.3386	
Do.	15 B	57	do.	6,165	11.692	104.1	.3286	
Do.	15 C	59	Mar. 28	5,907	10.915	105.0	.3340	
Do.	15 D	58	Mar. 27	5,697	10.666	105.8	.3270	
Do.	16 A	50	Mar. 22	5,175	10.333	105.1	.3090	Rejected in generalizing results. Steamed wood used.
Do.	17 A	49	Mar. 18	6,062	11.331	104.5	.3317	Do.
Do.	18 A	60	Mar. 28	6,484	12.089	105.4	.3292	
Do.	18 B	61	do.	6,070	11.220	105.8	.3310	
Do.	18 C	62	do.	6,283	11.894	104.8	.3265	
Do.	18 D	63	Mar. 29	6,385	11.800	105.4	.3326	
Do.	19 A	64	do.	6,747	12.636	104.9	.3297	
Do.	19 B	65	do.	6,784	12.828	105.2	.3257	
Do.	19 C	67	Mar. 30	6,274	12.072	105.8	.3180	
Do.	19 C	70	Mar. 31	6,460	12.072	105.9	.3273	
Do.	19 D	69	do.	6,481	11.891	106.4	.3318	
Red cedar	21 A	51	Mar. 23	4,958	8.986	104.4	.3423	Do.
Quaking aspen.	22 A	66	Mar. 30	4,342	8.403	103.9	.3222	
Do.	22 B	68	Mar. 31	4,309	7.945	105.8	.3320	
Red cedar	23 A	80	Apr. 11	4,580	8.716	106.3	.3202	
Do.	23 B	81	do.	2,920	5.909	106.9	.2990	Rejected in generalizing results.
Do.	23 B	82	Apr. 12	3,136	5.909	106.1	.3240	
Longleaf pine.	24 A	78	Apr. 11	7,178	12.887	106.3	.3390	
Do.	24 B	79	do.	6,816	12.527	106.3	.3315	
White pine.	25 A	71	Apr. 6	2,552	4.658	107.5	.3300	
Do.	25 B	72	do.	2,696	4.961	106.9	.3290	
Red spruce.	26 A	73	do.	4,090	7.570	106.9	.3273	
Do.	26 B	74	do.	4,508	8.230	106.5	.3330	
Beech.	27 A	83	Apr. 12	8,090	15.439	105.6	.3215	
Do.	27 B	84	do.	7,466	14.086	105.5	.3256	
Chestnut.	28 A	85	do.	5,617	10.901	105.9	.3148	
Do.	28 A	87	Apr. 13	5,720	10.901	106.9	.3179	
Do.	28 B	86	Apr. 12	4,800	9.460	104.9	.3132	
Do.	28 B	88	Apr. 13	4,901	9.460	106.9	.3138	
White oak.	29 A	89	do.	7,421	14.562	106.9	.3086	
Do.	29 A	92	Apr. 14	4,904	9.290	106.4	.3213	
Do.	29 B	90	do.	5,815	10.931	106.3	.3242	
White elm.	40 A	91	do.	4,422	8.353	106.5	.3220	
Do.	40 B	93	do.	3,832	7.860	106.3	.2970	Do.
Do.	40 B	94	Apr. 18	4,183	7.860	106.3	.3241	
Mockernut hickory.	41 A	95	do.	6,257	11.648	106.4	.3270	
Do.	41 B	96	do.	6,484	12.187	106.5	.3236	
Black gum.	42 A	97	Apr. 19	3,860	7.234	106.3	.3250	
Do.	42 B	98	do.	3,943	7.497	106.2	.3210	
White ash.	43 A	99	do.	4,924	9.707	105.7	.3109	
Do.	43 A	100	Apr. 20	4,722	9.707	107.4	.2934	Do.
Do.	43 A	102	Apr. 21	5,230	9.707	106.5	.3279	
Do.	43 B	101	Apr. 20	4,585	8.610	107.3	.3212	
Unknown wood, very light. {	44 A	113	Apr. 26	2,506	4.774	106.3	.3200	
Do.	44 B	114	do.	2,349	4.429	107.4	.3200	
Mai champah.	45 A	117	Apr. 27	3,597	6.668	108.4	.3220	
Do.	45 B	118	do.	3,519	6.634	107.1	.3207	
Unknown wood, heavier than water. {	46 A	115	Apr. 26	3,637	6.853	107.3	.3201	
Do.	46 B	116	do.	3,673	6.839	107.6	.3233	
Cocobola negra.	47 A	119	Apr. 27	3,347	6.232	106.9	.3253	
Do.	47 B	112	do.	3,303	6.226	105.9	.3245	

Walter Mulford

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GIFFORD PINCHOT, Forester.

WOOD DISTILLATION.

By W. C. GEER, *Expert.*

[NOTE.—The Forest Service has received many inquiries about the commercial distillation of hardwoods and softwoods and the quantity of the products obtained. It has been impossible to answer these inquiries by letter as fully as desired, and the growing need of a popular publication which would briefly state a few facts on wood distillation has been strongly felt. For this reason this circular has been compiled. It is not intended to be technical in nature or to contain the results of original investigations, but rather to furnish a few facts concerning the wood distillation industry as it now stands in this country. Circulars of a more technical character and dealing with concrete problems in wood distillation will follow.]

INTRODUCTION.

There are two distinct processes for obtaining valuable products from wood by distillation—destructive distillation and steam distillation. In the destructive process the wood fiber is broken down and new compounds are formed, but in the steam process this is not properly the case. In both processes the volatile compounds of the wood are vaporized.

In destructive distillation heat is applied below the wood-containing vessel, which has a comparatively small pipe as its only outlet. The heat vaporizes the volatile compounds, such as water and turpentine, and breaks down the nonvolatile compounds, such as cellulose and the wood gums; it forms a number of new compounds, usually of a simpler chemical nature, and these in turn are vaporized with the water and turpentine, leaving a residue of charcoal. The decomposition of the wood in this process is exceedingly complicated and is not yet fully understood.

In steam distillation, which is much simpler, the wood is chipped and placed in a closed receptacle into which steam is blown from a boiler, and the volatile compounds which are not chemically united with the rest are vaporized and carried out of the retort with the steam. Though in practice the wood is often so much overheated that the wood fiber is slightly decomposed, and though it is quite possible to carry the overheating so far that the process becomes one

of destructive distillation, it is nevertheless true that "steam distillation," as the term is technically used, signifies the separation of volatile products from wood with, at most, but little decomposition of the wood fiber.

With both these processes the vaporized compounds after leaving the retort pass through water-cooled tubes, where they are condensed into the crude liquors which after refining yield marketable products.

Different woods give different marketable products after distillation. Thus, the hardwoods—beech, birch, and maple—yield acetate of lime, wood alcohol, and charcoal, and longleaf pine yields turpentine, tar, pine oils, and charcoal. This difference in the products is due to the fact that pine woods are resinous, while hardwoods are nonresinous. From the point of view of products, therefore, it is necessary to distinguish between the kinds of wood used, as well as between the distillation processes.

DESTRUCTIVE DISTILLATION OF HARDWOOD.

Hardwood distillation has been an established industry in the United States for a number of years. The products already mentioned are wood alcohol, charcoal, and acetate of lime, each of which has important uses. The plants are located in the northern part of the United States, where, except for the Appalachian hardwood belt, the hardwoods are most common.

The woods used are largely beech, birch, and maple, with the last preferred. The wood is cut into cordwood lengths and allowed to season for a year. According to the best information, the amount of the products obtained from green wood and from ordinary dry wood is not different, cord for cord, but the higher water content of green wood dilutes the distillate and necessitates more fuel for the carbonization. Excessive seasoning will doubtless reduce the yield of valuable constituents. Body wood is better than slab wood. Very small wood, such as thin edgings, carbonizes so rapidly that it must be mixed with larger pieces. The problem of the destructive distillation of sawdust has not yet been satisfactorily solved.

APPARATUS.

Wood is heated or carbonized in three forms of apparatus: (a) In brick kilns, (b) in retorts, (c) in ovens.

The charring of wood is a process as old as civilization. In the early days the wood was charred under sod in the old charcoal kiln, which has been a familiar sight over a good part of the world. The modern charcoal kiln is so made that valuable vapors are condensed from the smoke, which in the old-fashioned kiln escaped into the air and were wasted. Kilns are now mainly used to produce charcoal for blast furnaces for pig iron. They are made of brick, with a circular base, and divided approximately into two semicircular sections. They hold each about 50 cords, and are charged and discharged by hand. The vapors are carried off into condensers, where the condensable ones are liquefied.

The name "retort" is given to a small form of cylindrical vessel holding about three-fourths of a cord. The retorts are set horizontally in brickwork, in pairs, each pair forming a "battery," and heated from beneath. They are filled and discharged from a single

door in front, which can be tightly fastened. The top of the battery is often tiled and serves as a drying floor for acetate of lime. The condensers are of copper, and are cooled by water. A "run," from charging to recharging, takes twenty-four hours.

The invention of the "oven" form of carbonizing vessel marked a distinct forward step in wood distillation. Oven kilns are made large enough to hold from two to four cars, which are run in on tracks, each loaded with about 2 cords of wood. They are usually fired separately, and the vapors pass over into the condensers either at the side or at the end. In other respects they resemble the "retorts."

PRODUCTS.

Four crude products are obtained from each of these forms of carbonizing vessels: (1) Charcoal, which remains in the vessel; (2) a noncondensable gas, which is carried off by suitable pipes; (3) an aqueous liquor known as "pyroligneous acid;" and (4) wood tar, which is condensed with the pyroligneous acid.

The charcoal is cooled differently in the case of each distilling vessel, though in all cases it is cooled for forty-eight hours. With kilns, it is allowed to cool before being removed; with the retorts, it is shoveled into drums or cans and sealed from the air; and with the ovens, the loaded cars are run out and closed in large coolers, which are similar in form to the ovens.

The gas from the kilns is piped back into the kiln furnaces, where it serves to carbonize the wood. The gas from retorts and ovens is burned under the boilers or under the retorts.

The pyroligneous acid and the tar run off together from the condensers into vats, where the tar settles. The pyroligneous acid is reddish brown in color and has a strong, characteristic, burnt-wood odor. The tar, when in thin layers, is dark brown in color, and has a bad odor. These two liquid products are refined by processes which in general are the same for each of the three forms of carbonizing apparatus. The processes differ somewhat, however, at the different plants.

Dissolved in the tar are some of the valuable compounds of the pyroligneous acid, while dissolved in the pyroligneous acid are some tarry bodies. Both liquids are distilled in order to concentrate the valuable substances, which are chiefly acetic acid and methyl, or wood, alcohol. The concentrated liquid containing the acetic acid and methyl alcohol is neutralized with lime and distilled from a "lime-lee" still, giving (1) a residue which upon evaporation yields gray acetate of lime, and (2) a distillate which upon refining yields the various grades of wood alcohol.

Some plants obtain a crude, brown, evil-smelling wood alcohol, of 82 per cent strength, which is sent to a refinery for further treatment; others obtain a 95 to 99 per cent product without color or unpleasant odor. Wood alcohol is ill-smelling only when impure as a result of incomplete refining.

Oven and retort plants which produce alcohol no purer than 82 per cent secure about the following average yields from wood distillation per cord of wood:

Charcoal.....	45 to 52 bushels.
Gray acetate of lime.....	180 to 225 pounds.
Wood alcohol, 82 per cent.....	8 to 10 gallons.

The lack of chemical supervision at the works makes statements of yield a little confusing, since wood alcohol and acetate of lime are variable in quality and the number of gallons and pounds may therefore actually represent products of quite different composition.

Kiln plants obtain about the following yield per cord of wood:

Charcoal.....	45 to 52 bushels.
Acetate of lime.....	90 to 150 pounds.
Wood alcohol, 82 per cent.....	4 to 6 gallons.

USE OF PRODUCTS.

These compounds have a variety of uses, which may be briefly mentioned. Charcoal is used in blast furnaces for the production of pig iron, in copper and sugar refineries, in the production of gunpowder, for fuel, etc. Wood alcohol is sold under a variety of trade names, such as "columbian spirit" and "colonial spirit." It is most widely used as a solvent in the production of shellacs and varnishes. It is also used in hat making, in perfumery, in the coal-tar dye industry, in manufacture of formaldehyde, and for mixing with grain alcohol to produce "denatured" or "industrial" alcohol. The acetate of lime is a gray, finely crystalline body, which is used in the manufacture of wood vinegar, acetic acid, many commercial acetates, acetic ether, acetone, and other products. (From the acetone may be produced iodoform and chloroform.)

A number of receipts for the preparation of denatured alcohol have been recently authorized by Congress and established by the Commissioner of Internal Revenue, so that denatured alcohol, with its due admixture of wood alcohol, is now a market article. The wood distillation plants now in existence in the United States are able to produce probably 30,000,000 gallons of wood alcohol annually.

Denatured alcohol is now a competitor of wood alcohol. At present the producers and refiners of wood alcohol are in suspense as regards the extent of the consumption of the product for denaturing purposes.

STEAM DISTILLATION OF HARDWOOD.

Several species of hardwood are distilled by steam in order to obtain valuable essential oils. Sweet birch, for example, yields "oil of wintergreen," an oil used in medicinal preparations. No thorough study has yet been made of this division of the subject, but it is known that a small industry is supported.

DESTRUCTIVE DISTILLATION OF YELLOW PINE.

The destructive distillation of yellow pine is carried on in the Southern States, where the distillation plants are so widely scattered that a statement of the location by States would mean but little.

The wood generally used is that of longleaf pine, from which turpentine and rosin are mainly obtained. At some plants, however, longleaf pine, shortleaf pine, Cuban pine, and others are indiscriminately used, but for the best results longleaf and Cuban pines are selected. The most valuable material is wood rich in resinous contents, or "fat," in which lightwood and stumps rank first, wood immediately under the "box faces" next, and slabs and other mill refuse last. Pine sawdust is not used for destructive distillation.

APPARATUS.

Iron or steel retorts are used, varying in capacity from 1 to 4 cords. They are either vertical or horizontal. The vertical retorts have their long axis upright, and are set singly in brickwork with suitable flues, usually with the openings for charging and discharging at the top and bottom. The firebox below is at one side, so that the heat goes around the outside of the retort itself. Few of these retorts are now in use.

The horizontal retorts are similar to those used in hardwood distillation. Though they differ as to form, all are cylindrical steel vessels set in batteries in brickwork and are charged and discharged through doors at one or both ends. The gases escape through pipes to copper condensers. The firebox is sometimes constructed to fire two retorts at a time, though usually but one.

PRODUCTS.

Though there are a number of methods which differ somewhat in results, the five products usually obtained are: (1) Charcoal; (2) a noncondensable gas; (3) light oils, which are often taken in two fractions, one of which is a crude turpentine; (4) tar, and (5) pyroligneous acid. At some plants the light oil vapor, which volatilizes easily, is led off into condensers with the gas and pyroligneous acid, while the tar, which is heavier, is drawn off at the bottom; at others, the entire volatile product is driven off through a pipe at the top and, after passing through the condenser, is separated into the crude turpentine and tar fractions.

There is no more uniformity in heating methods than in the form of the retorts. The run is thirty-six or forty-eight hours, or longer.

Charcoal which is to be sold is cooled in the retort, and that which is to be used for fuel is drawn hot and sprayed with water to prevent fire. The gas is allowed to run to waste or is burned under the retorts and boilers.

The pyroligneous acid from hardwoods contains the most valuable products, but that from pine, which has a strong odor and a reddish-brown color, is of such different composition that very little is done with it. The yield from a cord of pine wood is, according to the most widely accepted figures, not more than 3 gallons of 82 per cent wood alcohol and about 70 pounds of brown acetate of lime. The extraction of wood alcohol from pine wood is not at present on a commercial basis, and at the majority of plants the pyroligneous acid runs to waste.

The crude turpentine is a dark red oil with the bad odor associated with products of destructive distillation. After proper fractional distillation, it yields for market a nearly colorless turpentine, which has a distinctive odor.

The tar is sometimes refined far enough to produce a good quality of retort tar and to yield oils which, with the heavy distillates from the crude turpentine, make disinfectants, wood creosote, and a number of market articles.

The refining processes, which are largely secret, are not the same at all plants, while the products sold are far from uniform.

Since few plants operate under the same conditions, and since a number of products may be obtained from the tar and crude turpentine, it is difficult to estimate the amount of products obtained from yellow pine. Moreover, the wood itself varies widely in resinous content. Heavy, rich "lightwood" contains the largest quantities of turpentine and other oils, whereas other kinds of "lightwood" may yield but little. Sapwood yields the least. The following table shows as nearly as practicable the ordinary yields per cord of wood obtained in practice by the destructive process:

Refined turpentine.....	7 to 12 gallons.
Total oils, including tar.....	50 to 75 gallons.
Tar.....	40 to 60 gallons.
Charcoal.....	25 to 35 bushels.

USES OF PRODUCTS.

The turpentine is used as a second grade, inferior to gum turpentine. There are no recognized grades of destructively distilled turpentine, and the composition of the turpentine from different plants is not uniform. Formerly it was poorly refined; it is now made practically colorless. In the refining, certain heavy oils are obtained, which, when combined with similar heavy oils from the tar, are made into "pine oils," used as disinfectants, paint dryers, wood preservatives, etc. One of the uses for the tar is cable coating. The uses of the acetate of lime, in this case "brown acetate," have already been mentioned. The charcoal is burned at the plant or sold for fuel. The pyroligneous acid in its crude form is occasionally sold, although most of it goes to waste.

Several causes have led to many failures among plants of this kind. One of these was bad management. Men engaged in the business, without training or a knowledge of the market, expected an immediate demand for the products. Another cause was the use of inferior retorts, which in many cases were made of thin steel and so were quickly burned out. A third was lack of perseverance when difficulties arose.

STEAM DISTILLATION OF YELLOW PINE.

The plants which distil wood by the steam method are located in the yellow pine belt. In general, the wood is the same as that used for the destructive distillation of yellow pine, but is separated into classes. Steam plants use the richest wood that can be secured, since turpentine is the only valuable product, although the wood after extraction is used for fuel. The wood is divided into three classes: (1) The rich "lightwood," of which several grades are used; (2) stumps, which are also rich in turpentine; and (3) sawmill waste, which includes sawdust, butt cuts, and slabs. All wood must be "hogged" into chips before it is placed in the retorts.

APPARATUS.

Both vertical and horizontal retorts are successfully used. But the wood is treated by two different methods, one using superheated steam under low pressure and the other saturated steam under higher pressure.

With superheated steam a vertical retort is used, and the steam, before entering the retort, passes through a superheater, which raises its temperature high enough to readily volatilize the turpentine. From the condensers the distillates run into a separator.

For saturated steam several sorts of retorts are used, and the steam enters them directly from the boiler. There are a number of patented devices, the most important differences in which have to do with methods of charging and discharging. The fundamental idea, however, is to maintain a sufficient pressure of steam, throughout the run, to facilitate rapid extractions. A separator is used, as with superheated steam.

PRODUCTS.

The products of both processes are crude turpentine and water, in a separator tank, and chips left in the retort. The turpentine, which is lighter than water, floats on the surface and is easily drawn off, ready for refining. The chips, after drying a short time in the air, are suitable for fuel.

In order to obtain a market grade of turpentine, the crude product should be refined by distillation with steam in a copper still. As it comes from the retort its color is slightly yellow.

There is the same variety in methods used as in other kinds of wood distillation, and consequently the same lack of uniformity in the products. Much remains to be learned as to the best method of refining turpentine so as regularly to secure the best grades.

The amount of turpentine obtained from steam distillation varies widely. The wood itself varies greatly in richness. A conservative average per cord is given in the following table (the difference between stumps and "lightwood" is slight enough to be disregarded):

Lightwood:	
Refined turpentine.....	10 to 15 gallons.
Heavy oils.....	1 to 3 gallons.
Sawdust:	
Refined turpentine.....	2 to 4 gallons.
Heavy oils.....	$\frac{1}{2}$ gallon.

The refined turpentine is of reasonably uniform quality, is nearly colorless, has an agreeable odor, and has a fair market at a price somewhat below the market price for gum spirits of turpentine.

COMPARISON OF METHODS.

Comparing the steam methods with the destructive methods, although there is room for difference of opinion, it would seem that the steam distillation is open to the wider development. The successful destructive distillation plants are those which are run by men who have remained in the business long enough to establish their processes and methods and the markets for their products. Turpentine, the leading product, is probably produced less expensively by the steam method, and the steam apparatus necessary to handle a given quantity of wood per day, say 50 cords, is easier to operate.

There have been fewer failures in steam distillation than in destructive distillation, perhaps because it is of more recent development, and

because those promoting the enterprises have been able to profit by the mistakes of their predecessors. Yet many operators have failed, mainly because they had not familiarized themselves with the fundamental principles controlling the successful construction and operation of a plant.

The figures on pages 6 and 7 are not intended to compare yields by destructive and steam distillation from the same grade of wood, but simply the yields obtained by the two methods under actual conditions, where, in point of fact, very different grades of wood are used.

There is but scanty published information on the properties of the turpentine produced by these two processes in America, or on their actual value in the paint and varnish business. Up to the present these turpentine are merely competitors of "gum spirits."

Approved:

JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., *August 19, 1907.*

[Cir. 114]

O.



TYPICAL WESTERN YELLOW PINE TURPENTINE AREA IN ARIZONA.

Issued October 22, 1912.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE—BULLETIN 116.
HENRY S. GRAVES, Forester.

FOREST PRODUCTS LABORATORY SERIES.

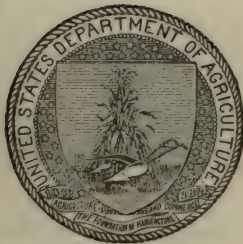
POSSIBILITIES OF WESTERN PINES
AS A SOURCE OF NAVAL STORES.

RECEIVED
OCT 30 1914

BY

H. S. BETTS,
Assistant District Forester.

Division of Forestry
University of California



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1912.

LETTER OF TRANSMITTAL

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE,

Washington, D. C., June 19, 1912.

SIR: I have the honor to transmit herewith a manuscript entitled "The Possibilities of Western Pines as a Source of Naval Stores," by Mr. H. S. Betts, Assistant District Forester, and to recommend its publication as Bulletin 116 of the Forest Service.

Very respectfully,

HENRY S. GRAVES, *Forester.*

Hon. JAMES WILSON,
Secretary of Agriculture.

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OCT 30 1914

Division of Forestry
University of CaliforniaPOSSIBILITIES OF WESTERN PINES AS A SOURCE OF
NAVAL STORES.

PURPOSE OF THE INVESTIGATION.

The constantly decreasing supply of southern yellow pine timber available for turpentine operations, and the steady demand for turpentine and rosin, suggest the utilization of western pines for the production of naval stores.

During 1911 the Forest Service conducted experiments on western yellow pine in Arizona and California,¹ and on both western yellow pine and piñon in Colorado, to determine the quantity of crude oleoresin which could be secured from these pines by the methods ordinarily employed in the turpentering of longleaf yellow pine in the Southeast. The field work was supplemented by laboratory analyses to determine the quality of the gum. The experiments in California were conducted under the supervision of C. Stowell Smith, assistant district forester.

ARIZONA EXPERIMENTS ON WESTERN YELLOW PINE.

(*Pinus ponderosa* var. *scopulorum*.)

DESCRIPTION OF EXPERIMENTAL AREAS.

The Arizona experiments were conducted on the Coconino and Tusayan National Forests, about 9 miles north of Flagstaff. Cups were hung on three different areas, chosen to represent average forest conditions, and designated A, B, and C, with 500 faces on each. These three areas combined made 28½ acres. In addition, 100 cups were hung on another area, D, for studying certain special problems. Figure 1 shows the location, altitude, and size of each of the experimental areas. The forest consisted of a pure stand of a variety of western yellow pine, running about 7,000 board feet to the acre. The surface of the ground was rolling and scattered over it were a large number of rocks of volcanic origin, commonly known as malpais rock. There was practically no undergrowth and but few trees below 12 inches in diameter breast high. Plate I shows a typical

¹ Experiments were started on lodgepole pine, sugar pine, and Douglas fir in California. The flow of gum was so small that the work was discontinued. During the season of 1912 the experiments on western yellow pine in California will be continued. The flow from digger pine, piñon pine, and Jeffrey pine will also be determined.

stand on one of the experimental areas. The number of cups per acre varied from 37 in area A to 87 in area C. The trees were divided into two classes, "yellow pine" and "black jack," and the dip from each class collected separately. The name "black jack" is commonly used for young western yellow pine with black, deeply furrowed bark and a comparatively dense crown. Later in life the bark becomes orange and smoother, and the tree is then called "yellow pine." The period in the tree's life at which the change takes place is varia-

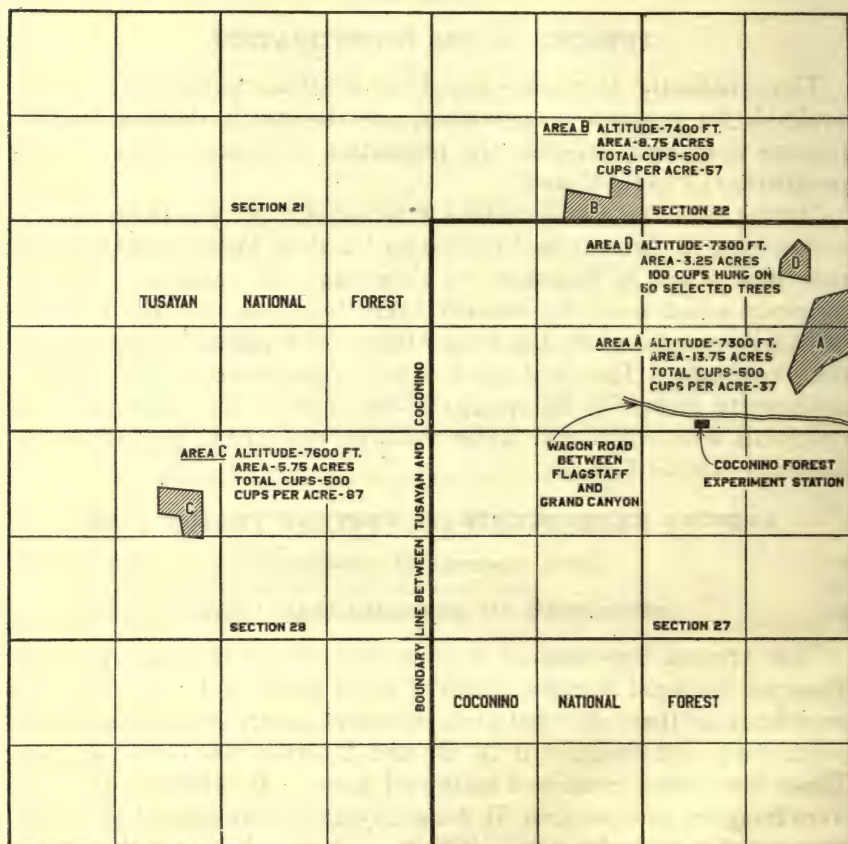


FIG. 1.—Location of experimental areas in Arizona.

ble, and it is fairly common to find black jack of considerably larger diameter than yellow pine growing close by. Table 1 shows the number and size of the tapped trees in each of the three areas, A, B, and C.

The average size of the trees in area A was greater than that in B or C. In area C the stand was denser and the trees more uniform in size than in the other two areas. Yellow pine predominated in areas A and B, while in C the stand was composed mainly of black jack.

Of the 388 yellow pines tapped, 89 had some defect, such as a fire-scarred butt, dead top, or large number of dead limbs. Of the 395 black jacks, only 15 were defective. The greatest proportion of defective trees was in area A among the large yellow pines.

TABLE 1.—*Number and size of tapped trees in the Arizona experiments; areas A, B, and C.*

Diameter, breast high.	Number of trees.								
	Area A.			Area B.			Area C.		
	Black jack.	Yellow pine.	Total.	Black jack.	Yellow pine.	Total.	Black jack.	Yellow pine.	Total.
Inches.	Number.	Number.	Number.	Number.	Number.	Number.	Number.	Number.	Number.
12.....	4	1	5	7	3	10	13	2	15
13.....	3		3	8	2	10	25	2	27
14.....	11	1	12	19	7	26	28	4	32
15.....	8	2	10	11	4	15	34	7	41
16.....	6	3	9	10	8	18	23	5	28
17.....	6	5	11	13	8	21	17	8	25
18.....	13	3	16	7	6	13	18	3	21
19.....	12	6	18	6	9	15	17	4	21
20.....	16	7	23	4	18	22	11	7	18
21.....	4	11	15	3	17	20	3	2	5
22.....	9	7	16	1	18	19	3	3	6
23.....	2	6	8	2	12	14	2	5	7
24.....	1	7	8		14	14	4	8	12
25.....	1	6	7	1	12	13	3	4	7
26.....	1	15	16		9	9		5	5
27.....		3	3		5	5		2	2
28.....	1	7	8	1	4	5			
29.....	1	11	12		4	4		1	1
30.....	2	6	8		4	4		1	1
31.....		7	7		3	3		2	2
32.....		5	5		3	3			
33.....		4	4					1	1
34.....		3	3						
35.....		3	3						
36.....		4	4		1	1			
37.....		3	3					1	1
38.....		1	1						
39.....		1	1						
40 and over.....		12	12						
Total.....	101	140	241	93	171	264	201	77	278

¹ One of these 45 inches in diameter.

Tapped trees on areas A, B, and C: Black jack, 395; yellow pine, 388; total, 783.

WEATHER CONDITIONS.

Table 2 shows the weather conditions throughout the Arizona experiments:

TABLE 2.—*Meteorological record, Fort Valley (Ariz.) experiment station, 1911.*

Month.	Temperature.			Precipitation.		Relative humidity.		
	Mean.	Maximum.	Minimum.	Rain.	Snow.	Mean.	Maximum.	Minimum.
	° F.	° F.	° F.	Inches.	Inches.	Per cent.	Per cent.	Per cent.
April.....	39.9	65.0	17.9	0.22	Trace.	51	90	20
May.....	47.9	72.2	21.9	Trace.		46	70	21
June.....	56.4	83.1	26.3	.22		40	74	16
July.....	60.7	83.8	30.4	5.71		69	97	26
August.....	61.4	84.9	41.2	2.76		59	92	25
September.....	56.8	79.2	34.5	3.47		61	100	20
October.....	44.7	70.3	12.6	2.68		54	100	17
Nov. 1-15.....	35.9	51.0	20.5	.22	Trace.			

The month of August shows the highest mean temperature (61.4° F.) and, as would be expected, the greatest yield of dip was during this month (see Table 3). Hardly any rain fell until July, when 5.71 inches was recorded. The three following months also had a substantial rainfall. The total precipitation from April 1 to November 15, 15.28 inches, was above the average for previous years. The mean humidity was greatest in July, and the driest weather in June. The mean temperature, humidity, and rainfall during the experiments were all below the average of similar observations made in Florida in connection with experiments there. In that part of Arizona in which the experiments were conducted the diurnal range of temperatures is much greater than in the turpentine regions of the Southeast, and a drop of from 35 to 40 degrees during the night is not at all uncommon. These conditions cause the gum to harden on the faces at night, and it takes a comparatively long period under the influence of the morning sun to start the flow again.

METHOD OF CONDUCTING EXPERIMENTS.

All trees of 12 inches or more breast-high diameter were tapped, except where they were so defective that they would not be used in a commercial operation. No cups were placed on trees less than 12 inches in diameter. One cup was placed on trees between 12 and 15 inches in diameter; two cups on trees between 15 and 24 inches; and three cups on trees more than 24 inches in diameter; but not more than three cups were hung on any one tree, no matter how large. On the other hand, less than the prescribed number of cups were hung in some cases because of defects in the tree.

The cups were of galvanized iron. In hanging them, the rough outer bark near the base of the tree was first smoothed off, and a concave-edged broadax was then driven in with a maul in order to make an incision for a galvanized-iron "apron." The apron was pushed tightly into the incision and the cup held below it by a nail driven into the tree, on which the weight of the cup rested. Plate II shows a "face" at the end of the season. The trees were chipped once a week with an ordinary turpentine "hack." An experienced turpentine hand from the Southeast was employed to do the chipping. While it was the intention to make the streaks one-half inch deep and to add one-half inch to the height of the face at each chipping, measurements taken at the end of the season showed the average depth of the streaks to be 0.68 inch and the average height 0.77 inch. This excess over the measurements originally planned was due to the fact that the workman had been trained to make a much wider and deeper cut. The cups were dipped every three weeks. Eight dippings were made on each area, the first on May 30, three weeks after the first streak was put on, and the last on October 31.



TURPENTINED "FACE" ON WESTERN YELLOW PINE (ARIZONA).

The gum was removed from the cups with a wooden paddle and collected in buckets. Each bucketful was weighed to determine the amount of dip collected.

These details are set forth to show that the experiment was conducted with the idea constantly in mind to make the operations similar to those of the Southeast, so that comparable results might be secured.

YIELDS.

COMPARISON OF YIELDS OF DIP IN ARIZONA AND FLORIDA

Table 3 shows the weight of crude gum or dip secured from the three experimental areas at the various dippings.

TABLE 3.—*Weight of dip obtained from western yellow pine in Arizona.*

Area A.		Area B.		Area C.	
Date of dipping.	Weight of dip.	Date of dipping.	Weight of dip.	Date of dipping.	Weight of dip.
1911.	Pounds.	1911.	Pounds.	1911.	Pounds.
May 30.....	233.75	June 1.....	151.75	June 6.....	171.25
June 20.....	397.75	June 22.....	320.50	June 27.....	323.50
July 11.....	435.75	July 13.....	407.00	July 18.....	366.50
Aug. 1.....	388.75	Aug. 3.....	372.50	Aug. 8.....	400.75
Aug. 22.....	465.25	Aug. 24.....	406.00	Aug. 29.....	417.50
Sept. 12.....	389.25	Sept. 16.....	362.25	Sept. 19.....	345.75
Oct. 3.....	335.75	Oct. 5.....	299.00	Oct. 10.....	255.00
Oct. 24.....	316.25	Oct. 26.....	205.25	Oct. 31.....	151.50
Total.....	2,862.50	Total.....	2,524.25	Total.....	2,431.75
Average per cup per week.....	.238	Average per cup per week.....	.210	Average per cup per week.....	.203

Total weight of dip, all areas, pounds, 7,817.50.

Average weight of dip per cup per week, pound, 0.217.

Each area of 500 cups chipped 24 times.

There was a total yield of 7,817.5 pounds of gum for the three areas combined (1,500 cups), or an average flow of 0.217 pound per cup per week. Experiments conducted by the Forest Service in Florida show the following yields of crude gum and scrape for the virgin crops:

TABLE 4.—*Weight of dip and scrape obtained from southern yellow pine in Florida, 1905.*

Crop designation.	Weight of dip.	Average weight of dip per cup per week.	Weight of scrape.
A.....	Pounds. 63,615.5	Pounds. 0.256	Pounds. 9,570
B.....	61,161.5	.246	7,650
C.....	62,587.0	.252	7,245
D.....	73,703.5	.297	8,888
Average.....	65,266.9	.263	8,338

Each crop 8,000 cups, chipped 31 times.

The average yield of dip from the four crops of 8,000 cups each was 65,267 pounds, or 0.263 pound per cup per week. This makes the yield from western yellow pine 82.5 per cent of that obtained from southern yellow pine. In making this comparison the same unit of time is used in both cases, that is, the flow per cup per week. In order that the yield per crop from Arizona and Florida may be on a comparable basis the Arizona yield of 7,817.5 pounds from 1,500 cups may be expressed as the equivalent of a crop of 8,000 cups or 41,700 pounds. This is 64 per cent of the average yield secured in the Florida experiments during the season of 1905. In the Arizona experiments the first chipping was done May 8 and the last dipping made October 31. Had the cups arrived earlier they could have been hung in April and the season lengthened to 26 weeks instead of 24.

COMPARISON OF YIELDS OF SCRAPE IN ARIZONA AND FLORIDA.

Table 5 shows the amount of scrape collected from the experimental areas and the percentage of faces bearing scrape in sufficient quantity to collect.

TABLE 5.—*Scrape obtained from western yellow pine in Arizona.*

Area.	Weight of scrape.	Faces bear- ing scrape.
	<i>Pounds.</i>	<i>Per cent.</i>
A	523.75	87
B	314.75	55
C	288.25	56
All areas	1,126.75	66

Of the 1,500 faces on the three experimental areas 990 had sufficient scrape on them to warrant removing it. The total yield of 1,126.75 pounds of scrape from 1,500 faces chipped 24 times is equivalent to an average yield of 0.031 pounds per face per week. Reduced to a common basis the Florida experiments (see Table 4) show a yield of 0.034 pound of scrape per face per week. Hence the average production of scrape per face in the Arizona experiments was 91 per cent of that in the Florida experiments, if the yields are compared for equal periods of time.

The total yield of 1,126.75 pounds of scrape from 1,500 faces is equivalent to a yield of 6,014 pounds of scrape from a crop of 8,000 faces. Table 4 shows that the weight of scrape obtained in the Florida experiments averages 8,338 pounds per crop of 8,000 faces. The yield from western yellow pine is therefore 72 per cent of that obtained from southern yellow pine when the comparison is made on the basis of the whole season.

Area A produced considerably more scrape than areas B or C. This is accounted for in part by the fact that about one-third of the faces on A were worked for six weeks in the preceding year (1910).

COMPARISON OF YIELDS OF DIP AND SCRAPE FROM BLACK JACK AND YELLOW PINE.

The yields of dip and scrape from both black jack and yellow pine are shown in Table 6:

TABLE 6.—*Weight of dip and scrape obtained from black jack and yellow pine.*

BLACK JACK.

Area.	Number of cups.	Weight of dip.		Weight of scrape.	
		Total.	Per cup per week.	Total.	Per face.
		<i>Pounds.</i>	<i>Pound.</i>	<i>Pounds.</i>	<i>Pounds.</i>
A.....	175	1,063.5	0.253	211.50	1.21
B.....	140	754.0	.224	89.75	.641
C.....	332	1,535.0	.193	192.00	.578
All areas.....	647	3,352.5	.216	493.25	.762

YELLOW PINE.

A.....	325	1,799.00	0.231	312.25	0.961
B.....	360	1,770.25	.205	225.00	.625
C.....	168	896.75	.222	96.25	.573
All areas.....	853	4,466.00	.218	633.50	.742

On areas A and B the black jacks gave a larger flow per cup than the yellow pines, while on C this was reversed. But including all trees of both classes the average yield per cup was practically the same. The production of scrape on area A was considerably larger per face for black jack than for yellow pine; on B and C the production per face was about the same for both classes of timber; taking all areas, therefore, the production of scrape ran slightly higher for black jack than for yellow pine.

COMPOSITION OF DIP.

The results of 22 analyses of samples of crude gum are shown in Table 7. These analyses show only the proportions of volatile oil, rosin, water, and chips in the crude gum. Ordinary turpentine from the southern yellow pines consists almost entirely of pinene. A further analysis¹ of the volatile oil from gum obtained in Arizona shows it to be mostly pinene with a small proportion of a higher boiling terpene not found in ordinary turpentine. There is no apparent reason why Arizona turpentine can not be used commercially with entire satisfaction. The same is true of the Arizona rosin.

¹ Forest Service Bulletin 119, An Examination of the Oleoresins of Some Western Pines, by A. W. Schorger.

Considering the pure dip, free from water and chips, there is little variation in the proportions of rosin and turpentine. The average of all the samples, 77.9 per cent rosin and 22.1 per cent turpentine, is practically the same as that of a number of samples of dip from southern yellow pine. The proportion of water and chips in the Arizona dip is considerably lower than in samples of dip collected from turpentine operations in the Southeast. This is to be expected, because in the Southeast the chipping is done as piecework, and the chipper in his hurry frequently neglects to place a guard over the cups, even when he is required to use a guard, which is but seldom. The comparatively few cups hung in Arizona enabled the work to be carefully supervised. Table 7 indicates a slightly higher proportion of turpentine during the beginning and latter part of the season than in the summer months. There is practically no difference in the proportions of rosin and turpentine in gum from black jack and yellow pine.

TABLE 7.—Results of laboratory analyses on samples of dip from western yellow pine, Arizona.

Area from which sample was taken.	Date of collection.	Class of timber.	Analysis of samples.					
			As taken.				Free from water and chips.	
			Water.	Chips.	Rosin.	Turpentine.	Rosin.	Turpentine.
			Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
A.....	May 30	Yellow pine.	1.0	2.2	73.9	22.9	76.3	23.7
A.....	July 11	do.....	3.5	3.9	71.7	20.9	77.4	22.6
A.....	Aug. 22	do.....	3.8	0.2	75.3	20.7	78.4	21.6
A.....	Oct. 3	do.....	5.7	0.6	71.8	21.9	76.6	23.4
A.....	June 20	Black jack..	2.3	0.8	73.4	23.5	75.8	24.2
A.....	Aug. 1	do.....	9.5	1.4	67.1	22.0	75.3	24.7
A.....	Sept. 12	do.....	4.9	3	74.7	20.1	78.8	21.2
A.....	Oct. 24	do.....	3.4	0.2	70.2	26.2	72.8	27.2
B.....	June 22	Yellow pine.	2.0	3.1	74.4	20.5	78.7	21.3
B.....	Aug. 3	do.....	5.0	2.2	75.6	17.2	81.5	18.5
B.....	Sept. 16	do.....	5.1	1.7	74.0	19.2	79.4	20.6
B.....	Oct. 26	do.....	4.4	1.0	71.5	23.1	75.6	24.4
B.....	June 1	Black jack..	1.9	2.8	73.9	21.4	77.5	22.5
B.....	July 13	do.....	3.7	2.0	75.8	18.5	80.4	19.6
B.....	Aug. 24	do.....	3.5	1.3	75.6	19.2	79.8	20.2
B.....	Oct. 5	do.....	4.3	1.5	73.4	20.8	77.9	22.1
C.....	June 6	Yellow pine.	1.1	0.9	75.3	22.7	76.8	23.2
C.....	Aug. 8	do.....	1.7	0.6	79.9	17.8	81.7	18.3
C.....	Oct. 10	do.....	6.2	0.8	72.7	20.3	78.2	21.8
C.....	June 6	Black jack..	1.3	1.7	75.6	21.4	77.9	22.1
C.....	Aug. 8	do.....	4.9	0.2	75.3	19.6	79.3	20.7
C.....	Oct. 10	do.....	6.0	0.7	72.8	20.5	78.0	22.0
Average of all samples..			3.9	1.37	73.8	20.9	77.9	22.1

CONDITION OF TAPPED TREES.

At the time the cups were hung, the trees were examined at the butt to determine if they were infested with boring beetles. Very few of the trees showed signs of insect attack, but, because of the thick, rough bark the examination told but little, since the evidences of beetle infestation are not noticeable except in severe cases. At the

conclusion of the experiment also the faces on a number of trees on each area were examined. Of 276 trees inspected, the faces on 116 showed indications of borers. These indications, consisting of small deposits of finely divided wood, were generally found on the lower part of the face near the apron at the junction of the inner bark and sapwood. Whether or not the trees were infested with borers before the cups were hung is not known. It was noted that practically all the trees which showed signs of borers also contained holes made by woodpeckers, doubtless in search of the insects.

Of 634 faces examined on the three areas 5 were found to be dry and 26 partially dry.

EVAPORATION FROM CUPS.

The rate of evaporation from the cups was determined by exposing cups half full of fresh gum to the action of the air and weighing them at regular intervals. The samples of gum were secured by taking small amounts from as many of the cups as necessary on the first or second day after a fresh streak had been put on. Two samples were taken from each area after each dipping. As dip is collected every three or four weeks in commercial operations, the loss in weight during the first four weeks is the significant figure in the evaporation tests. Forty-eight evaporation samples were used. Of these, 14 showed no loss at all in weight for the first four weeks; the remaining 34 samples showed losses ranging from 1.5 to 10.5 per cent of the original weight. The average loss in weight for all the samples was 3 per cent. The gum as exposed contained turpentine, rosin, water, and chips. The loss by evaporation was, of course, made up of turpentine and water. The average loss of turpentine by evaporation from the cups in the Arizona experiments was therefore less than 3 per cent. No evaporation figures are available for southeastern operations.

SPECIAL PROBLEMS INVESTIGATED.

Area D contained 50 selected trees 15 inches or more in diameter breast high. Two cups were hung on each tree—one on the north side and one on the south side. In 25 of the trees the first streak was put on so as to bring the peak of the face 3 inches above the apron. In the remaining 25 trees the first streak was put on so as to bring the peak 18 inches above the apron. Of the 50 selected trees about half were black jack and half yellow pine. The 3-inch and 18-inch faces were placed on black jack and yellow pines in about equal proportions. The rough outer bark between the peak and the apron was smoothed off in all of the trees. The trees were chipped every week in the ordinary manner. The cups were dipped every week and weighed on the third, fifth, and seventh day after each chipping throughout the season by means of a small portable balance. The dip from the

low and high faces was kept separate and typical samples chosen for analysis.

COMPARISON OF YIELDS FROM NORTH AND SOUTH FACES.

The total yield for the 50 cups on the north side of the trees from the first dipping on June 3 to the last dipping on November 3 was 242.6 pounds and for the south cups 266.2 pounds. These weights show a 9 per cent greater flow on the south side of the trees than on the north. Figure 2 shows the average yield per week for both the north and south faces of each of the 50 trees arranged in order of the yields from the south faces. Twenty-seven south cups yielded more than the corresponding north cups, while 17 north cups showed an excess over the corresponding south cups. The remaining six trees had about the same flow for both cups. The diagram shows

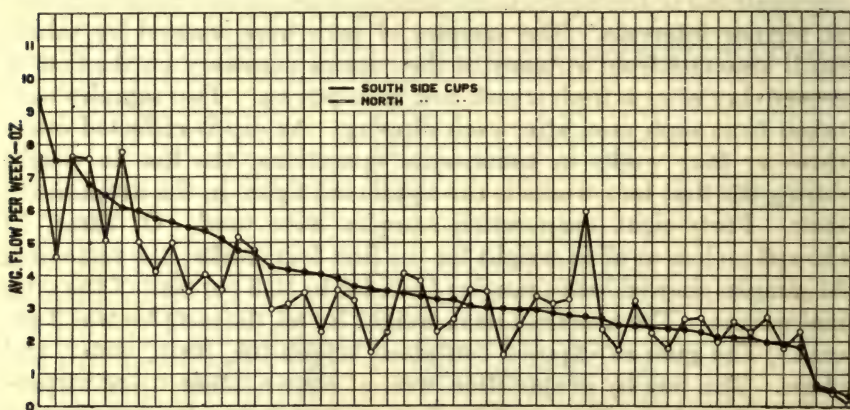


FIG. 2.—Comparison of flow from north and south faces of each of fifty trees.

the tendency of faces on the same tree to give the same yields. Trees having an exceptionally good or exceptionally poor flow generally show it in both faces.

COMPARISON OF DIP FROM HIGH AND LOW FACES.

While there was an attempt to determine what greater loss there would be, if any, from evaporation from the higher faces, it was found to be impossible to select trees with comparable flows. As far as it was possible to carry out the experiment, the indications were that the proportions of rosin and turpentine in the dip were not affected by the height of the face, and this would seem to show that there was no appreciable difference of evaporation on faces with a difference in height of 15 inches. Table 8 gives the composition of dip from the two heights of faces. The results of the analyses given in this table are very similar to those given in Table 7.

TABLE 8.—Composition of dip from 3-inch and 18-inch faces.

Date of collection.	3-inch faces.		18-inch faces.	
	Rosin.	Turpentine.	Rosin.	Turpentine.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
June 10.....	78.9	21.1	78.2	21.8
	76.5	23.5	78.4	21.6
July 21.....	80.0	20.0	79.2	20.8
	78.7	21.3	78.5	21.5
Sept. 1.....	80.0	20.0	78.6	21.4
	78.4	21.6	78.9	21.1
Oct. 13.....	78.3	21.7	77.0	23.0
	77.0	23.0	78.3	21.7
Average.....	78.5	21.5	78.4	21.6

EFFECT OF TEMPERATURE ON WEEKLY FLOW.

Figure 3 shows the average flow per cup for the 50 trees on area D for each week and the corresponding average temperature. The

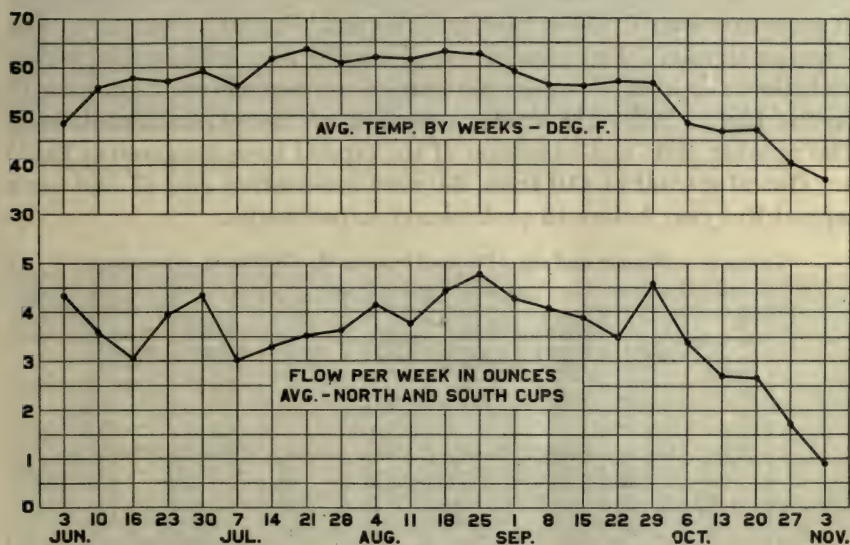


FIG. 3.—Relation between temperature and rate of flow of gum.

flow increased or decreased with increase or decrease of temperature with few exceptions. The effect of cool weather in checking the flow is especially marked toward the end of the season.

RATE OF FLOW DURING WEEK.

It is well known that gum flows most rapidly just after a fresh streak and that the rate of flow decreases until at the end of a week it has become so slow as to necessitate rechipping. Data on the variation in rate of flow were secured by weighing the north and south cups on 10 trees on the third day after each chipping throughout the season. The sum of the weights for each tree was compared with

the weight of the total flow for the season from the same trees. An average of the results shows that 73 per cent of the weekly flow occurred in the first three days. This ratio varied from 65 per cent to 78.6 per cent in the 10 test trees.

CALIFORNIA EXPERIMENTS ON WESTERN YELLOW PINE.

(*Pinus ponderosa*.)

DESCRIPTION OF AREA AND METHOD OF EXPERIMENT.

The California turpentine experiments on western yellow pine were conducted on the Sierra National Forest about 2 miles from North Fork, Madera County. Nine hundred and sixty-eight cups were hung on about 160 acres, which included several ridges extending from a main ridge, and ranged between 3,500 and 4,500 feet in elevation. No cups were hung on the few trees in the small canyons between the ridges, and a few large trees with extra thick bark also were left out. The stand was very open, and consisted principally of mature western yellow pine with a sprinkling of young pines and some scrub oak. The ground was generally free from undergrowth, with the exception of occasional patches of bear clover from 6 to 18 inches high. Plate III shows a portion of the experimental area. The number, size, and condition of the tapped trees are given in Table 9. Out of a total of 410 trees, 64 were fire-scarred and 47 had been tapped the year before in preliminary experiments.

TABLE 9.—*Number and size of tapped trees in the California experiments.*

Diameter breast high.	Total trees.	Trees with fire- scarred butt or "cat face."	Trees tapped previous year.	Diameter breast high.	Total trees.	Trees with fire- scarred butt or "cat face."	Trees tapped previous year.
Inches.	Number.	Number.	Number.	Inches.	Number.	Number.	Number.
12	8	3	2	29	13		
13	10	3		30	16	1	2
14	15	1	1	31	13	3	1
15	10	2	2	32	11	2	1
16	20	4	1	33	12	2	
17	13	2		34	6	1	
18	21	1	4	35	7	2	
19	20	4		36	4	0	
20	29	4		37	2	1	
21	22	3	1	38			
22	25	3	2	39	1		
23	19	3		40	2	1	
24	34	6	10	41			
25	29	4	5	42			
26	18	4	4	43	2	0	
27	12	1	2				
28	16	3	1	Total..	410	64	47

The same procedure in hanging the cups and chipping was followed as in the Arizona experiments, except that the chipping was done by forest rangers and guards instead of by an experienced turpentine hand. The first streak was put on July 7 and the last dipping made November 1; during this period of approximately 16 weeks the trees were chipped 16 times and dipped 8 times.



FIG. 1.—TYPICAL WESTERN YELLOW PINE TURPENTINE AREA IN CALIFORNIA.



FIG. 2.—TYPICAL WESTERN YELLOW PINE TURPENTINE AREA IN COLORADO.

WEATHER CONDITIONS.

Table 10 shows the weather conditions during the California experiments, including three months preceding and one month following. The mean temperature on the area from June 1 to November 30 was a few degrees lower than the mean temperature recorded for the Florida ¹ experiments from March 17 to November 2. The diurnal range is much greater than in Florida. The total rainfall on the California area for the period of the experiments amounted to only 2.16 inches.

TABLE 10.—*Meteorological record, Northfork (California) ranger station, 1911.*

Month.	Temperature.				Precipitation.	
	Mean.	Maximum.	Minimum.	Average range during 24 hours.	Rain.	Snow.
	° F.	° F.	° F.	° F.	Inches.	Inch.
March.....	50.1	84	30	25	12.44	0
April.....	50.2	79	30	29	4.25	0
May.....	56.3	87	32	34	1.18	0
June.....	66.8	103	46	37	Trace.	0
July.....	79.5	108	52	39	0	0
August.....	72.5	101	50	39	0	0
September.....	63.7	98	42	39	.35	0
October.....	61.2	94	32	43	.3	0
November.....	54.0	90	24	45	1.51	0
December.....	41.5	76	15	29	1.58	0

YIELDS.

Table 11 shows the weights of gum secured from the area at the various dippings. The average yield of 0.281 pound per cup per week is slightly greater than that in the Florida experiments (0.263 pound; see p. 9), but it should be kept in mind that the California experiments were not begun until July and that had they been started earlier in the season this average might have been lower. Seventeen faces were chipped at monthly intervals during December, January, and February. A yield of 16 pounds of dip was obtained, which shows that gum continues to flow throughout the winter.

TABLE 11.—*Weight of dip obtained from western yellow pine in California (968 cups).*

Date of dipping.	Weight of dip.	Date of dipping.	Weight of dip.
	Pounds.		Pounds.
July 18.....	630	Oct. 19.....	578
July 18.....	384	Nov. 1.....	490
Aug. 16.....	702		
Sept. 5.....	610	Total.....	4,366
Sept. 15.....	418	Average per cup per week.....	0.281
Sept. 27.....	554		

¹ Forest Service Bulletin 40, A New Method of Turpentine Orcharding, by Charles H. Herty.

The total weight of scrape obtained from the area at the end of the season amounted to 287 pounds. This is equivalent to a yield of 0.018 pound of scrape per face per week, somewhat less than obtained in the Florida experiments (see Table 4).

COMPOSITION OF DIP AND SCRAPE.

The results of analyses of two samples of crude gum and one sample of scrape are given in Table 12. The proportion of turpentine in the California gum, as shown by these samples, is less than in the Arizona gum (see Table 7). A further analysis of the volatile oil from California western yellow pine shows that it contains a considerably smaller proportion of pinene than ordinary turpentine, and contains another oil not found in ordinary turpentine. It is, however, probable that turpentine from California western yellow pine will prove of commercial value. Its use is recorded in California at the close of the Civil War, when turpentine operations in the Southeast were practically at a standstill. The operations in California were very crude, however, and were discontinued as soon as naval stores from the Southeast became available.

TABLE 12.—Results of laboratory analyses on samples of dip and scrape from western yellow pine, California.

Material.	Date of collection.	Composition of samples.					
		As taken.				Free from water and chips.	
		Water.	Chips.	Rosin.	Turpentine.	Rosin.	Turpentine.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Dip.....	Oct., 1910.	1.2	0.8	79.5	18.5	81.1	18.8
Do.....	Oct., 1911.	1.0	1.6	81.1	16.3	83.2	16.7
Scrape.....	Oct., 1910.	1.1	.8	84.9	13.2	86.4	13.4

EVAPORATION FROM CUPS.

The loss from the cups by evaporation was tested by weighing four cups approximately half full of fresh gum on September 7 and again on December 1. The average loss in weight was 2.8 per cent.

COLORADO EXPERIMENTS ON PIÑON.

(*Pinus edulis.*)

DESCRIPTION OF AREA AND METHOD OF EXPERIMENT.

The turpentine experiments on piñon were conducted on the Montezuma National Forest. The cups were placed on a tract 2½ miles southeast of Mancos, representative of average conditions in the piñon stands of southwestern Colorado. The elevation is 7,200 feet; the surface is rolling with a gentle slope to the southwest;

the soil is adobe, very thin, and underlaid by the Mancos shales. The trees were of various sizes; those of medium size generally occurred in groups, while the old trees usually stood alone. There was heavy underbrush, which made the work of placing the cups very difficult. The many defects in the trees, such as gnarled, twisted, and partially dead butts, and low limbs, further complicated the problem. It was necessary to place some of the cups 2 or 3 feet from the ground because of these conditions. One hundred and twenty-three cups were hung on 4 acres, approximately 31 cups to the acre. Ninety-five trees were tapped; of these 67 had 1 cup and 28 had 2 cups. The diameter of the tapped trees ranged from 7 to 26 inches about 1 foot above the ground. Plate IV shows a single piñon on the edge of the experimental area.

The method of placing the cups was the same as in the Arizona experiments, except that no attempt was made to limit the minimum diameter of tapped trees or the number of cups placed on any one tree. The chipping was done by a ranger who soon became accustomed to handling a hack.

WEATHER CONDITIONS.

Table 13 shows the weather conditions at Mancos while the turpentine experiment was in progress. Mancos is at approximately the same elevation as the piñon area.

TABLE 13.—*Weather record at Mancos, Colo., June to October, 1911 (U. S. Weather Bureau).*

Month.	Temperature.		Precipitation.
	Maximum.	Minimum.	
	°F.	°F.	Inches.
June.....	86	33	1.92
July.....	83	43	5.39
August.....	88	38	1.82
September.....	83	36	3.32
October.....	68	14	3.63

YIELD.

The weight of dip secured from the piñon area at the various dippings is shown in Table 14.

TABLE 14.—*Weight of dip obtained from piñon pine in Colorado (123 cups).*

Date of dipping.	Weight of dip.	Date of dipping.	Weight of dip.
	Pounds.		Pounds.
June 30.....	25.0	October 13.....	50.0
July 21.....	50.0	October 31.....	12.0
August 11.....	71.0		
September 1.....	75.0	Total.....	366.5
September 22.....	63.5	Average per cup per week.....	.147

The total yield of 366.5 pounds represents the dip secured from 123 cups from June 9, when the first streak was put on, to October 31, the date of the last dipping, a period of approximately 20 weeks. The average yield of 0.147 pound per cup per week is about two-thirds as much as secured from western yellow pine in Arizona and a little over half the average yield of southern yellow pine in Florida operations.

COMPOSITION OF DIP.

Table 15 gives the results of analyses on five samples of piñon dip.

TABLE 15.—*Results of laboratory analyses on samples of dip from piñon, Colorado.*

Date of collection.	Rosin.	Turpentine.
	<i>Per cent.</i>	<i>Per cent.</i>
June 30.....	81.3	18.7
July 15.....	78.3	21.7
Aug. 11.....	81.0	19.0
Sept. 1.....	78.3	21.7
Sept. 23.....	78.0	22.0
Average..	78.4	20.6

NOTE.—Per cents of rosin and turpentine are calculated for gum free from water and chips.

The proportion of turpentine in the piñon gum was slightly less than in the gum from Arizona western yellow pine (see Table 7). The turpentine from piñon is composed mainly of pinene and in this respect is similar to turpentine from southeastern pines, but it also contains another oil of quite different physical properties. The turpentine, however, could probably be used for industrial purposes.

COLORADO EXPERIMENTS ON WESTERN YELLOW PINE.

(*Pinus ponderosa* var. *scopulorum*.)

DESCRIPTION OF EXPERIMENTAL AREAS.

The turpentine experiments on western yellow pine in Colorado were conducted on two areas, one on the Montezuma National Forest and the other on the San Juan. The Montezuma area is about 7 miles from Mancos, on a southwestern slope at an elevation of about 7,800 feet. The area was lightly cut over several years ago, and some of the largest trees were removed; but it still contains trees of all ages and is a representative stand for the locality. The soil is thin and rocky. One hundred and twenty-five cups were hung on an area of 6 acres. Sixty-eight trees were tapped, ranging from 13 to 32 inches in diameter breast high. The first streak was put on June 10.

The San Juan area is about 6 miles north of Durango on a fairly steep slope at an elevation of about 7,500 feet. The area contained considerable undergrowth. Plate III, fig. 2, shows a typical view on the San Juan area. One hundred and twenty-five cups were hung on 62



MATURE ISOLATED PIÑON ON TURPENTINED AREA IN COLORADO.

trees, ranging in size from 14 to 42 inches diameter breast high. Because of adverse weather conditions, weekly chippings were skipped three times during the course of the experiment, so that instead of 21 chippings from June 16 to November 4 there were but 18. Considerable rain fell while the experiment was in progress. In Durango, which is some 2,500 feet lower than the turpentine area, the precipitation from June 1 to October 31 was 17.08 inches. The greater elevation and the topographic features of the experimental area tend to cause a much greater precipitation there than in Durango. During the last few weeks of the experiment there were several snow flurries and periods of cold weather with freezing at night.

The method of hanging the cups and conducting the work on both areas was the same as in the Arizona experiments, except that the chipping was done by rangers. The trees used in the Colorado and Arizona experiments were of the same species and variety.

YIELDS.

Table 16 shows the weights of dip obtained from western yellow pine on the Montezuma and San Juan areas.

TABLE 16.—Weights of dip obtained from western yellow pine in Colorado.

Montezuma area.		San Juan area.	
Date of dipping.	Weight of dip.	Date of dipping.	Weight of dip.
	<i>Pounds.</i>		<i>Pounds.</i>
July 2.....	52.75	July 8.....	34.75
July 24.....	68.5	July 29.....	58.5
Aug. 12.....	89.0	Aug. 19.....	75.0
Sept. 2.....	87.0	Sept. 9.....	66.75
Sept. 23.....	79.0	Oct. 8.....	55.75
Oct. 14.....	75.0	Nov. 4.....	27.5
Nov. 1.....	18.0		
Total.....	469.25	Total.....	318.25
Average per week per cup.....	.188	Average per week per cup.....	.127

The average flow per cup per week on the Montezuma area was 0.188 pound, and on the San Juan 0.127 pound; in both cases lower than the average of 0.217 pound secured from the same pine in the Arizona experiments. This difference is perhaps due in part to the fact that the chipping in Arizona was done by an experienced turpentine hand. On the Montezuma area the greater regularity in carrying on the experiment accounts, at least partly, for the higher yield as compared with that from the San Juan area.

SUMMARY OF RESULTS.

The Arizona experiments show a yield of resin from western yellow pine (*Pinus ponderosa scopulorum*) about four-fifths as great as that obtained from southern yellow pines (*Pinus palustris* and *Pinus*

heterophylla) in average operations in Florida ¹ if the comparison is based on the same period of time for both. Weather conditions in Arizona, however, will allow only a 24 or possibly a 26 weeks' season as against a 30 or 35 weeks' season in the Southeast, so that when the yields for an entire season are compared, western yellow pine shows a production about two-thirds as great as that from southern yellow pine. The average proportions of rosin and turpentine in the gum were about the same as in gum from the southeastern pines, and the turpentine had a composition much like that from the Southeast.

The California experiments on western yellow pine, carried on over a period of 16 weeks from July 7 to November 1, show an average rate of flow slightly greater than that in the Florida experiments. Had the work in California been started earlier, it is probable that the weekly average would have been lower, as a smaller yield is to be expected early in the season. The season of flow in California will undoubtedly be as long as in Arizona, and probably longer. The composition of the volatile oil obtained by distilling the gum from California differs from that of ordinary turpentine somewhat more than does the Arizona turpentine, but the oil probably will be satisfactory for industrial purposes.

Piñon (*Pinus edulis*) in Colorado had a rate of flow slightly over half that of the Florida pines for a 20-weeks period, from June 9 to October 31. The volatile oil from the piñon gum differs somewhat from ordinary turpentine, but is probably suited to industrial use.

PROBLEMS WHICH CONFRONT COMMERCIAL DEVELOPMENT.

In considering the possibilities of commercial turpentine operations on western pines the problem of labor is one of the first that presents itself. In Arizona, Mexicans constitute a large part of the laboring class, but they are totally unfamiliar with turpentine work. Negro turpentine hands could be brought in from the Southeast, but their transportation would be costly. A few negro hands might be secured to teach the Mexicans, but whether the results would be satisfactory is, of course, unknown. In California both Indian and white labor is available in many timbered portions that have turpentine possibilities; here also the chippers would have to be taught how to use a hack.

The shorter season in Arizona, as compared with that of the Southeast, and the comparative severity of the winters in the timbered parts of the State might make it necessary to discontinue operations entirely for a few months during the winter. This would necessitate the

¹ As a basis of comparison for the yields obtained in the West, the results of experiments made by the Forest Service in Florida several years ago are used. (See Bulletin 90, Relation of Light Chipping to the Commercial Yield of Naval Stores, by Charles H. Herty.)

reorganization of the operating force each spring, with a great many attendant difficulties. The California experiments were not started until June, so that the time when the gum begins to flow is not yet known. The flow continued in California longer than in Arizona. Temperature records taken on the California and the Arizona areas show a considerably higher average temperature in California. The diurnal range of temperature is greater in California than in Arizona.

Western yellow pine timber generally grows in open stands free from underbrush. In the majority of stands there would be but little, if any, more difficulty in moving the crude gum than in the Southeast. On rough ground burro pack trains might be used. Two small kegs or buckets holding about 150 pounds of dip could be slung on each animal.

The number of cups that can be hung on an acre of average western yellow pine compares favorably with many areas that are now being turpented in the Southeast. The western trees are larger than most of the southeastern ones. The bark of the western pines is, however, thicker and rougher, and it will be necessary to remove the outer bark before hanging cups or chipping the trees; and this, of course, means the expense of an extra step not necessary in southeastern operations. Such work can be done by the use of a broad ax or heavy spade-shaped tool with a cutting edge.

The cost of securing turpentine rights in the Southeast is constantly rising, and it is likely that turpentine stumpage could be leased at lower rates in the West.

At present the turpentine and rosin used in the West is shipped from the Gulf States. The advantage of cutting out a two or three thousand mile haul to western markets is evident.

The commercial success of turpentine operations in the Southwest will be doubtful until they have been tried on a commercial scale. Nearly as much turpentine and rosin was obtained from western yellow pine, and the amount of timber available for turpentine operations in the Southeast is constantly diminishing. These two facts make it reasonable to suppose that turpentine operations in the large tracts of virgin pine timber of the West will, in time, be justified.

Issued January 11, 1913.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE—Bulletin 119.
HENRY S. GRAVES, Forester.

FOREST PRODUCTS LABORATORY SERIES.

AN EXAMINATION OF THE OLEORESINS
OF SOME WESTERN PINES.

RECEIVED

DEC 1 1914

BY

A. W. SCHORGER,
Chemist in Forest Products.

Division of Forestry
University of California



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DEPARTMENT OF AGRICULTURE

FOREST SERVICE

WASHINGTON, D. C.

FOREST SERVICE LABORATORY REPORT

ANALYSIS OF THE GROWTH
OF SOIL WEATHERING PLANT

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U. S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE



LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE,
Washington, D. C., July 24, 1912.

SIR: I have the honor to transmit herewith a manuscript entitled "An Examination of the Oleoresins of Some Western Pines," by A. W. Schorger, chemist in forest products, and to recommend its publication as Bulletin 119 of the Forest Service. The samples of oleoresins examined were collected by forest officers in Districts 3 and 5; the chemical work was performed at the Forest Products Laboratory, maintained in cooperation with the University of Wisconsin, at Madison, Wis.

Respectfully,

HENRY S. GRAVES,
Forester.

HON. JAMES WILSON,
Secretary of Agriculture.

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DEC 1 1914

Division of Forestry
University of CaliforniaEXAMINATION OF THE OLEORESINS OF SOME
WESTERN PINES.

GENERAL PROPERTIES AND COMPOSITION OF OLEORESINS.

A wound in the sapwood of a growing conifer induces, to a greater or less extent, the flow of a liquid resembling honey in appearance and consistency. This liquid, called oleoresin, consists of a solution of resin acids in a volatile oil.

Turpentine is the trade name applied to the volatile oil; it is also sometimes called "oil of turpentine" or "spirits of turpentine." In Europe the term "oil of turpentine" is invariably used to denote the volatile constituent, while "turpentine" alone signifies the oleoresin. In this country the oleoresin is commercially known as "gum" or "crude turpentine," while "dip" and "scrape" are terms commonly used to signify, respectively, the portions dipped from the receptacles used in turpentine operations or scraped from the scarified faces of the trees.

The turpentine, or volatile oil, is separated from the oleoresin by distillation, and the residue in the still, after being freed from trash, is the colophony or "rosin" of commerce.

Turpentine, from the chemist's standpoint, consists of a mixture of hydrocarbons having the empirical formula $C_{10}H_{16}$, called terpenes. American turpentine consists largely of the terpene α -pinene, with smaller amounts of β -pinene¹ and camphene.² French turpentine agrees closely with American turpentine in its physical and chemical properties.

Colophony or rosin is a hard, vitreous, amorphous body, breaking with a conchoidal fracture and varying in color from black to light amber. It consists of monobasic resin acids, which usually can be obtained in a crystalline state by appropriate treatment.

Knowledge of the resin acids, their empirical formula, constitution, and properties is in a very confused state. A large number of resin acids have been described and held to be distinct bodies, but it seems probable that the majority of these are modified forms of one or more typical acids. American colophony is very generally considered to contain from 80 to 95 per cent abietic acid. The physical properties

¹ Schimmel & Co. Report, April, 1908, pp. 99-100.

² Ibid., October, 1897, p. 68.

of abietic acid, such as melting point, optical rotation, and crystalline form, depend upon the methods by which the crystalline acid has been isolated;¹ a statement as to the method of isolation is especially necessary when melting points are given. The empirical formula of abietic acid has long been disputed. Numerous investigators have advanced the formula $C_{10}H_{28}O_2$,² but the weight of evidence is in favor of the formula $C_{20}H_{30}O_2$.³

PURPOSE OF THE STUDY.

Because of the rapid decrease in the supply of longleaf pine and Cuban pine available for the production of naval stores, the Forest Service conducted tapping experiments on several species of western pines to determine their value as a source of turpentine and rosin.⁴ The oleoresins of these species were examined to determine to what extent they agree with the similar product obtained from the southern pines. The investigation was purely a chemical one, and no practical experiments were made to ascertain what value the volatile oils would have in the paint and varnish industries. While no one of the oils from the western species agrees entirely with standard gum turpentines, there is no reason to conclude that the oils from several of the species examined will not serve for various industrial purposes.

KINDS OF OLEORESINS EXAMINED.

The oleoresins examined were from western yellow pine (*Pinus ponderosa* Laws.) grown on the Coconino National Forest in Arizona, and from western yellow pine, digger pine (*Pinus sabiniana* Dougl.), lodgepole pine (*Pinus contorta* Loud.), and sugar pine (*Pinus lambertiana* Dougl.) grown on the Sierra National Forest in California. These samples were secured in the fall of 1910. During 1911 the same species were tapped systematically from May to October. The oleoresins representing the collections of the entire period were analyzed also, and the results were entirely in accord with those previously obtained. Therefore, except in a few instances, the results of the later analyses are not given. However, during the year 1911, piñon pine (*Pinus edulis* Engelm.) was tapped on the Montezuma National

¹ Compare Schkateloff, *Moniteur Scientifique*, pp. 217-227, vol. 22, 1908, and Leskiewicz, *Jour. f. prak. Chem.*, p. 412, vol. 189, 1910.

² Compare Tschirch, *Die Harze und die Harzbehälter*, 2d ed., pp. 670-671, 1906; Mach, *Monatsh. für Chemie*, p. 186, vol. 14, 1893; *ibid.*, p. 627, vol. 15, 1894; Easterfield and Bagley, *Jour. Chem. Soc.*, p. 1233, vol. 85, 1904; Tschirch and Studer, *Archiv. d. Pharm.*, p. 495, vol. 241, 1903; *ibid.*, p. 523; *ibid.*, p. 3, vol. 245, 1907.

³ Compare Valente, *Atti della R. Accad. dei Lincei*, p. 13, vol. 1 (4), 1884; Fahrion, *Chem. Rev. über Fett- und Harz-Ind.*, p. 237, 1898; Levy, *Zeit. f. angew. Chem.*, p. 1740, vol. 18, 1905, and *Ber. Deutsch. Chem. Ges.*, p. 3043, vol. 39, 1906; Vesterberg, *Ber. Deutsch. Chem. Ges.*, p. 121, vol. 40, 1907; Koritschoner, *Zeit. f. angew. Chem.*, p. 642, vol. 20, 1907; and Leskiewicz, *Jour. f. prak. Chem.*, p. 412, vol. 189, 1910.

⁴ The results of the tapping experiments are given in Forest Service Bulletin 116, "Possibilities of Western Pines as a Source of Naval Stores," by H. S. Betts.

Forest, Colorado, and the analyses made on this species are included. A brief description of the samples upon which the analyses reported in this bulletin were made is here given.

Western yellow pine.—The oleoresin which was obtained from the Sierra National Forest was secured from trees tapped September 13 to October 12, 1910, which grew at an altitude of 4,000 feet. The sample amounted to about 50 pounds. The oleoresin of this species (var. *P. ponderosa scopulorum* Engelm.) sent from the Coconino National Forest in Arizona was obtained from trees tapped August 1 to September 15, 1910. Two dippings were made at intervals of three weeks. About 80 pounds of oleoresin, representing a mixture of both dippings, were shipped. The trees tapped grew at an altitude of 7,300 feet.

Digger pine.—The oleoresin was secured from trees which grew at an altitude of 2,700 feet and were first tapped August 19, 1910. The material shipped amounted to about 50 pounds.

Sugar pine.—The sample was from trees which grew at an altitude of 5,800 feet; they were tapped from September 13 to October 12, 1910. The sample weighed about 5 pounds.

Lodgepole pine.—The trees, at an altitude of 6,000 feet, were tapped from September 13 to October 12, 1910, and the sample weighed about 5 pounds.

Piñon pine.—The oleoresin was secured from trees tapped from June 9 to October 31, 1911. The sample, amounting to 50 pounds, represented the "dip" secured from 5 dippings at 3-weeks intervals.

In all cases the oleoresins were collected by a cup system. The samples were shipped in closed tin cans within from 1 to 4 weeks after collection. A further period, varying from 1 to 2½ months, elapsed before the examination of the samples was begun, during which time the material was retained in the shipping cans. The metal of the cans showed no indications of attack by the crude gum.

METHODS OF EXAMINATION.

OLEORESIN.

The general examination of all the oleoresins was the same. From 350 to 400 grams of the crude resin were distilled with steam in a tared 1.5-liter balloon flask, with a Kjeldahl connecting bulb tube placed between the flask and condenser to prevent any rosin from being carried over mechanically. The rosin remaining in the flask was kept heated until all the water had been expelled, care being taken that the temperature did not exceed 145°. When bubbles ceased to form on the surface of the rosin, on agitation, the removal of the water was considered complete. The rosin was then strained through two layers of fine muslin, and that which adhered was com-

pletely removed by extraction with chloroform in a Soxhlet apparatus. The weight of trash obtained, subtracted from the combined weight of trash and rosin after drying, gave the weight of the rosin. The percentage of water originally in the oleoresin was obtained by difference.¹

VOLATILE OIL.

The oil for examination was secured by distillation with steam. About 500 grams of this oil were fractionally distilled, using a 12-inch Hempel column, and the specific gravity and refractive index of the fractions were determined with the Westphal balance and Abbe refractometer. The detailed method of fractionation is given in Bulletin 105² of the Forest Service.

The methods employed in the chemical examination of the volatile oils will be discussed in connection with the determination of their various constituents.

ROSIN.

The grade of the rosin was determined by comparison with the standard rosin cubes used in the naval stores trade.

The specific gravity was determined by means of a salt solution of such density that a fragment of the rosin, free from cracks and bubbles, remained suspended in it, neither rising nor sinking. The solution was maintained at a temperature of 15° C. and its specific gravity determined with a hydrometer. The specific gravity of the rosin was considered equal to that of the salt solution.

The acid number was obtained by dissolving from 1 to 1.5 grams of rosin in 95 per cent alcohol and titrating the solution directly in the cold, with a N/2 alcoholic potassium hydroxide solution using phenol-phthalein as the indicator. The number of milligrams of potassium hydroxide required to neutralize 1 gram of the rosin represents the acid number.

The saponification number was determined by treating from 1 to 1.5 grams of powdered rosin with 25 cubic centimeters of N/2 alcoholic potassium hydroxide solution. After heating the solution for one-half hour on a steam bath, and then cooling, the excess potassium hydroxide was determined by titration with a N/2 sulphuric acid solution using phenol-phthalein as the indicator. The saponification number represents the number of milligrams of potassium hydroxide neutralized by 1 gram of rosin.

Rosin does not contain esters but contains a small proportion of bodies having the nature of lactones. The measure of the latter

¹ The water may be conveniently determined directly as follows: 100 c. c. of dry redistilled turpentine are added to a given weight of oleoresin (approximately 100 grams) and 100 c. c. of turpentine distilled over by direct heat. The distillate containing all the water is collected in a graduated funnel. On standing a short time the distillate separates into two layers and the amount of water is read off.

² "Wood turpentine, their analysis, refining, and composition," by L. F. Hawley.

represents the ether number and is obtained by subtracting the acid number from the saponification number.

The methods of isolating the resin acids will be discussed under the several species, since each species usually requires a different treatment. The composition of the resin acids was checked by means of their silver salts, which were obtained in the following way: A weighed amount of the resin acids was dissolved in alcohol and the solution was exactly neutralized with alcoholic caustic soda. The sodium salt was then decomposed by adding the calculated amount of silver nitrate dissolved in a small amount of water. The precipitate of silver salt was filtered off with a suction pump and the residue washed with water, alcohol, and finally with ether. The precipitate was dried at 60° for half an hour and then allowed to stand a day over sulphuric acid.

ABBREVIATIONS.

The following abbreviations are used:

$d_{15^{\circ}}$ or "specific gravity" = specific gravity at 15° compared with water at 15°.

$n_{D15^{\circ}}$ or "index of refraction" = index of refraction at 15°

$\alpha_{D20^{\circ}}$ = optical rotation at 20° in a 100 mm. tube.

$[a]_D$ = specific rotation.

RESULTS OF EXAMINATIONS BY SPECIES.

Pinus Ponderosa Laws (California).

LITERATURE.

The oleoresin of *Pinus ponderosa*, or western yellow pine, evidently has never been subjected to a careful examination. Sadtler¹ examined an oil coming supposedly from *Pinus ponderosa*. However, since the oil he examined had the general properties of normal heptane, it must have come from either *Pinus sabiniana* or *Pinus jeffreyi*, which, so far as known, are the only species that yield heptane. Confusion between the species might readily occur, because *Pinus jeffreyi* so closely resembles *Pinus ponderosa* that it is often considered a variety. Wenzell² had examined in his laboratory a sample of oleoresin from *Pinus ponderosa* obtained from Virginia, Nev., and stated that the odor, specific gravity, and boiling point corresponded with common oil of turpentine. The material examined in the forest products laboratory does not corroborate this statement.

¹ Am. Jour. Pharm., pp. 176-180, vol. 51, 1879.

² Pharm. Rev., pp. 165-172, vol. 18, 1900.

EXAMINATION OF THE OLEORESIN.

The oleoresin of the western yellow pine from the Sierra National Forest, Cal., had a specific gravity of 1.0290 at 15°. Following is the average composition shown by four analyses:

	Per cent.
Volatile oil.....	18.5
Rosin (grade WW, or highest grade).....	79.5
Chips, needles, etc.....	.8
Water (by difference).....	1.2
Total.....	100.0

An analysis of scrape showed the following composition:

	Per cent.
Volatile oil.....	13.2
Rosin (grade H, or seventh grade).....	84.9
Chips, needles, etc.....	.8
Water by difference.....	1.1
Total.....	100.0

EXAMINATION OF THE VOLATILE OIL.

PHYSICAL PROPERTIES.

The specific gravity of the volatile oil was 0.8625; the index of refraction, 1.4772; and the specific rotation, $[a]_D = -14.39^\circ$. On fractionation of 500 grams of oil the results given in Table 1 were obtained:

TABLE 1.—Fractional distillation of the volatile oil of *Pinus ponderosa* from the Sierra National Forest, Cal.

Temperature (corrected).	Distillate.		Specific gravity at 15°.	Index of refraction at 15°.	Specific rotation at 20°.
	Observed.	Cumulative.			
° C.	Per cent.	Per cent.			
164.0-168.2	5.01	5.01	0.8578	1.4740	-13.81°
168.6	9.94	14.95	.8595	1.4750	-13.70
169.0	9.36	24.31	.8601	1.4752	-13.17
169.2	9.20	33.51	.8606	1.4753	-12.92
170.5	10.45	43.96	.8608	1.4754	-12.73
171.0	10.71	54.67	.8612	1.4757	-12.98
171.6	10.28	64.95	.8615	1.4760	-13.33
172.4	9.69	74.64	.8619	1.4762	-14.17
175.0	10.36	85.20	.8621	1.4763	-17.15
179.5	7.04	92.24	.8631	1.4775	-24.13
179.5+	7.74	99.98	.9186	1.5063	+ 0.86

IDENTIFICATION OF THE CONSTITUENTS.

α -pinene.—The first fraction was examined for α -pinene. Redistillation over metallic sodium gave two equal fractions boiling between 156° and 164° and 164° and 165° C. The portion boiling between 156° and 164° gave a precipitate with nitrosyl chloride by Wallach's¹ method. The crystalline pinene nitrosochloride sepa-

¹ Annalen, p. 251, vol. 245, 1888.

rated very reluctantly from the mother liquor. The nitrosochloride, after several precipitations from chloroform with methyl alcohol, melted at 103° ; the melting point of its nitrolpiperidine was 118° . These results prove the presence of *l*- α -pinene.

β -pinene.—The fractions boiling between 168.2° and 172.4° were redistilled several times and about 60 per cent (based on the original 500 grams) boiled between 165° and 170° . After seven fractionations, the final one made over metallic sodium, 200 grams were obtained having the following properties: Boiling point, 166.6° to 167.6° ; specific gravity, 0.8670; index of refraction, 1.4762; specific rotation, $[\alpha]_D = -15.33^{\circ}$. This fraction was then examined for β -pinene by Wallach's¹ method, which is as follows: The volatile oil was emulsified with 500 c. c. of water and oxidized with 467 grams of potassium permanganate and 100 grams of caustic soda in 5.5 liters of water with careful cooling. The resulting liquor was distilled with steam and 137 grams of oil recovered. After filtration from the manganese sludge and evaporation to two liters, a stream of carbon dioxide being continuously passed through the solution, a copious precipitate of crystalline sodium nopinate was obtained. After being washed with water and then dried the crude sodium nopinate amounted to 13.8 grams. This gives a yield of 21.9 per cent of the sodium salt based on 63 grams of oxidized oil. The yield is comparatively high and shows the purity of the β -pinene fraction. The nopinic acid was liberated with dilute sulphuric acid. After two crystallizations from water it melted at 126° .

Limonene.—The fractions of the original oil boiling above 172.4° and the residues boiling above 170° obtained from the fractionation of the β -pinene fraction were examined for limonene. By repeated distillation two 1-rotatory fractions were obtained, as follows: (1) Boiling point, 174° to 175.7° ; specific gravity, 0.8621 at 15° C.; (2) boiling point, 175.7° to 178.6° ; specific gravity, 0.8608 at 15° C. Fraction 1 when dissolved in glacial acetic acid and treated with a solution of bromine in acetic acid gave a tetrabromide after a small amount of water had been added. The crystalline tetrabromide melted at 104° after several crystallizations from ethylacetate. Fraction 2 was redistilled over sodium, diluted with ether, and treated with dry hydrogen chloride. A dihydrochloride was obtained which melted at 50° C. The results show the presence of *l*-limonene.

EXAMINATION OF THE ROSIN.

A "water white" sample of the rosin had the following properties: Specific gravity, 1.068; acid number, 148.8; saponification number,

¹ Annalen, p. 228, vol. 356, 1907.

161.3; ether number, 12.5. The specific rotation of the rosin was determined for an alcoholic solution, using the formula $[a]_D = \frac{100a}{l p d}$ in which

$a = -0.66^\circ$, the optical rotation in a 1-decimeter tube at 20°C .

$l = 1$, the length of the tube in decimeters.

$p = 6.22$, the number of grams of the substance in 100 grams of the solution.

$d = 0.8239$, the density of the solution.

Therefore, the specific rotation, $[a]_D = -12.88^\circ$.

The coarsely powdered rosin was digested with twice its weight of alcohol containing 25 per cent concentrated hydrochloric acid. The residue yielded about 90 per cent of crude crystals. These crystals, recrystallized from acetone until white, melted at 159° to 160° ; the specific rotation, $[a]_D$, is computed from the following values:

$$\begin{array}{lll} a = -2.61^\circ & p = 4.06 & [a]_D = -78.44^\circ \\ l = 1 & d = 0.8196 & \end{array}$$

When allowed to crystallize slowly from dilute acetone and examined under the microscope, the crystals had the exact crystalline form figured by Schkateloff,¹ Mach,² and Levy³ for abietic acid. By rapid crystallization they have the form of triangular plates. This form can be readily obtained by dissolving the abietic acid crystals in acetone and adding water to the solution until it is turbid; as it cools, a mass of crystals immediately forms.

In order to confirm the presence of abietic acid the silver salt of the resin acid was prepared in the manner described on page 11, and its silver content determined. The results of the analysis were:

	Per cent.
0.1396 gram silver salt gave 0.0365 gram silver	=26. 15
.1120 gram silver salt gave .0295 gram silver	=26. 34
Mean	=26. 25

Silver abietate, $\text{Ag}(\text{C}_{20}\text{H}_{20}\text{O}_2)$, requires 26.37 per cent of silver.

Schkateloff's⁴ method of precipitating the resin acids works very well for the rosin of *Pinus ponderosa*. It consists in saturating an alcoholic solution of the rosin with sulphur dioxide. The resin acids, which crystallize out on cooling, are white and much purer than those obtained by crystallizing from alcohol and hydrochloric acid. No "bleaching" of the color of the alcoholic solution, which Schkateloff noted, was observed, even though the gas was passed in for two hours.

Some of the original rosin was dissolved in acetone and allowed to stand in an ice box overnight. The crystals obtained were purified

¹ Monit. Scientif., p. 218, vol. 22, 1908.

² Monatsh. f. Chem., p. 629, vol. 15, 1894.

³ Berichte, p. 3044, vol. 39, 1906.

⁴ Monit. Scientif., p. 223, vol. 22, 1908.

by recrystallization from the same solvent. They softened at 145° , melted at 155° , and were perfectly liquid at 159° .

The fact that the resin acids begin to soften several degrees below their melting point and are not completely liquid until several degrees above it, even when slowly heated, may account, in part, for the diverse melting points recorded. In this work the first appearance of liquefaction has been taken as the melting point.

A portion of the original rosin was subjected to distillation at reduced pressure. The distillate passing over between 245° and 265° C. at 22 mm. solidified to a hard, pale amber mass. After a second crystallization from acetone, snow-white crystals were obtained; these softened at 145° , melted at 150° to 151° , and became completely liquid at 156° . Dissolved in alcohol, they gave the specific rotation, $[a]_D = -54.29^{\circ}$, computed from the values:

$$\begin{array}{ll} a = -0.72^{\circ} & p = 1.63 \\ l = 1 & d = 0.8137 \end{array}$$

It is important to note that the crystals obtained in the presence of hydrochloric acid melted at 159° to 160° , those obtained from acetone alone melted at 155° , and those obtained from the rosin distilled under reduced pressure melted at 150° to 151° . Schkateloff¹ claims that the various acids described in the literature are but different modifications of abietic (sylvic) acid, and that, according to the treatment given, the acid varies in melting point and crystalline form. His opinion is in part confirmed by the results given in this bulletin.

CONCLUSIONS.

From the distillation data it has been estimated that the volatile oil of *Pinus ponderosa* from California contains about 5 per cent *l*- α -pinene, 60 to 70 per cent *l*- β -pinene, and 20 to 25 per cent *l*-limonene. The rosin contains about 90 per cent abietic acid.

Pinus Ponderosa scopulorum Engelm. (Arizona).

EXAMINATION OF THE OLEORESIN.

Two analyses of the oleoresin of the Rocky Mountain form of the western yellow pine (*Pinus ponderosa scopulorum*) from the Cocalino National Forest, Ariz., gave the following average:

	Per cent.
Volatile oil.....	19.6
Rosin (grade M, or fourth grade).....	75.0
Chips, needles, etc.....	3.8
Water (by difference ²).....	1.6
Total.....	100.0

¹ Monit. Scientif., pp. 217-227, vol. 22, 1908.

² Determined directly, 1.85 per cent.

EXAMINATION OF THE VOLATILE OIL.

PHYSICAL PROPERTIES.

The oil had these properties:¹ Specific gravity, 0.8639 at 15°; index of refraction, 1.4727 at 15°.

The physical constants of the fractions obtained on distillation are shown in Table 2.

TABLE 2.—Fractional distillation of the volatile oil of *Pinus ponderosa scopulorum* from the Coconino National Forest, Ariz.

Temperature (corrected).	Distillate.		^d _{15°} Specific gravity at 15°.	N _{D15°} Index of refraction at 15°.
	Observed.	Cumulative.		
° C.	Per cent.	Per cent.		
157.6–158.5	2.03	2.03	0.8586	1.4686
159.5	9.69	11.72	.8621	1.4701
159.9	9.97	21.69	.8620	1.4702
160.1	10.21	31.90	.8620	1.4705
160.9	10.06	41.96	.8620	1.4706
161.7	9.39	51.35	.8620	1.4711
162.6	9.97	61.32	.8623	1.4714
165.2	9.12	70.44	.8626	1.4722
170.2	10.50	80.94	.8626	1.4735
173.0	5.94	86.88	.8615	1.4752
177.1	6.25	93.13	.8593	1.4758
183.3	2.19	95.32	.8576	1.4769
183.3+	4.60	99.92		

The original oil, and especially the last fractions, had a pleasant orange-like odor.

IDENTIFICATION OF THE CONSTITUENTS.

α-pinene.—The first fractions of the oil, when tested for *α-pinene*, readily yielded a nitrosochloride when treated with nitrosyl chloride; the purified nitrosochloride melted at 103°.

β-pinene.—The fractions boiling between 160° and 170° were examined for *β-pinene* by oxidation with potassium permanganate in the manner previously described. A small amount of nopinic acid (about 0.75 per cent) was obtained, which melted at 125°; this proved the presence of *β-pinene*.

Limonene.—The fractions boiling above 170° were repeatedly fractionated and two fractions were separated. These had the following properties: (1) Boiling point, 175° to 176°; specific gravity, 0.8588; (2) boiling point, 176° to 177°; specific gravity, 0.8580. Fraction 1 was distilled over sodium, dissolved in ether and treated with dry hydrogen chloride. A dihydrochloride was obtained, which melted at 50°. Fraction 2 was treated with bromine. The

¹ Two oils obtained from separate areas and representing the collection for the season 1911 had the following properties: (1) specific gravity, 0.8654 at 15°; index of refraction, 1.4723 at 15°; specific rotation, $[\alpha]_D = +13.03^\circ$; (2) specific gravity, 0.8672 at 15°; index of refraction, 1.4729 at 15°; specific rotation, $[\alpha]_D = +12.86^\circ$.

resulting tetrabromide, after several crystallizations from ethyl acetate, melted at 104.5° , the melting point of limonene tetrabromide.

Totally anhydrous reagents, it was found, prevented the precipitation of limonene tetrabromide. The limonene fraction dissolved in glacial acetic acid and treated with bromine gave no trace of a crystalline precipitate after standing in a cool place for two days. When a few drops of water were added, until a faint turbidity was produced, a copious precipitate of limonene tetrabromide soon followed.

Although this species is a variety of *Pinus ponderosa*, there are marked differences in the properties of the two oils, as shown by chemical analysis and inspection of the distillation curves (figs. 1, 2, and 3). All the oils of *Pinus ponderosa* examined were *l*-rotatory, while those of *Pinus ponderosa scopulorum* were *d*-rotatory. The former consisted largely of β -pinene and the latter of α -pinene.

EXAMINATION OF THE ROSIN.

After digestion of the rosin on the steam bath with a mixture of alcohol and concentrated hydrochloric acid, the crystalline residue was recrystallized from 95 per cent alcohol and gave a yield of about 90 per cent of abietic acid. Purified by repeated crystallizations from acetone, the abietic acid melted at 159° .

The original rosin, grade WW, had a specific rotation, $[\alpha]_D = -30.95^{\circ}$, determined from an 8.29 per cent alcoholic solution. The purified crystals in an alcoholic solution gave the specific rotation $[\alpha]_D = -88.02^{\circ}$ from the values:

$$\begin{array}{ll} a = -1.36^{\circ} & p = 1.90 \\ l = 1 & d = 0.8132 \end{array}$$

For further confirmation of the presence of abietic acid, the silver salts were analyzed, with the following results:

	Per cent.
0.3203 gram silver salt gave 0.0834 gram silver.....	=26.04
.0600 gram silver salt gave .0159 gram silver.....	=26.50
.1922 gram silver salt gave .0504 gram silver.....	=26.22
Mean.....	=26.25

Silver abietate, $\text{Ag}(\text{C}_{20}\text{H}_{29}\text{O}_2)$, requires 26.37 per cent silver.

It will be noted that this abietic acid is strongly *l*-rotatory, and it is probable that the lower rotations of other authors is due to a mixture of *d*- and *l*-acids. For the specific rotation of abietic acid Dieterich¹ gives -48° ; Haller,² -53° ; Mach,³ -69.96° ; Mead and Kremers,⁴ -53.42° ; Leskiewicz⁵ (for *l*-sylvic acid), -102.85° . Caillot⁶ observed, however, that the optical rotation decreased with

¹ Dissertation, Berne, 1883.

² Berichte, p. 2167, vol. 18, 1885.

³ Monatsh. f. Chemie, p. 641, vol. 14, 1894.

⁴ Proc. Am. Pharm. Ass'n, p. 198, vol. 41, 1893.

⁵ Jour. f. prak. Chem., p. 416, vol. 189, 1910.

⁶ Bull. Soc. Chim., p. 387, vol. 21, 1874.

the degree of heating and with the concentration of the solution. Acids isolated from the oleoresin of *Pinus sabiniana* and not heated above 60° gave -95.82° for the specific rotation, which approaches the rotation of the *l*-sylvic acid for which Leskiewicz found $[a]_D = -102.85^\circ$.

CONCLUSIONS.

From the distillation data it has been estimated that the volatile oil of *Pinus ponderosa scopulorum* contains 60 to 70 per cent *d*- α -pinene, about 5 per cent β -pinene, and 20 to 25 per cent limonene. The rosin contains about 90 per cent abietic acid.

Pinus Sabiniana Dougl.

LITERATURE.¹

The volatile oil obtained from the resinous exudation of the digger pine (*Pinus sabiniana*), or coming apparently from this species, has been the cause of considerable dispute. That a hydrocarbon, normal heptane, should occur in a growing plant was a source of wonder to phyto-chemists. Semmler (⁹), in the face of all the investigations that have been carried out on this oil, thinks there is a strong possibility that the material so far examined may have been adulterated with a petroleum hydrocarbon, and recommends the careful examination of the volatile oil obtained from authentic samples of oleoresin.

The question has been further complicated by contradictory statements as to what species of pine really furnished heptane. Wenzell (¹⁴), who first discovered "abietene," or heptane, reported it as coming from *Pinus sabiniana* and his later paper (¹⁵) is quite conclusive on this point. Sadtler (⁴) examined an oil having all the properties of "abietene," but coming supposedly from *Pinus ponderosa*.² Trimble (¹¹) states that *Pinus jeffreyi* was considered the main source of "abietene," although *Pinus sabiniana* and *Pinus ponderosa* may have furnished samples of "abietene." The papers of Wenzell (¹⁵) and Blasdale (¹) would indicate that both *Pinus sabiniana* and *Pinus jeffreyi* ³ yield heptane. The thorough chemical investigations by Thorpe (¹⁰), Schorlemmer and Thorpe (⁸), and Venable (¹²), as well as the work of Venable (¹³) and Grissom, and Young (¹⁶), left little doubt but that the oil from *Pinus sabiniana* is true normal heptane.

¹ The small numbers in parentheses refer to the bibliography on pp. 21-22.

² This seems erroneous, since the volatile oil from *Pinus ponderosa* consists of terpenes. (See pp. 12-13 of this paper.)

³ The oleoresin from *Pinus jeffreyi* has not been examined by the author, but it is hoped that this may be done later.

EXAMINATION OF THE OLEORESIN.

The crude oleoresin which was examined had a characteristic orangelike odor, and white crystals of resin acid had separated out. The specific gravity of the original material was 1.0265; an 8.45 per cent alcoholic solution gave for the optical rotation, $a_{D20} = -1.26^\circ$. The volatile oil was removed by distillation with steam, and the average of two analyses was as follows:

	Per cent.
Volatile oil.....	11.4
Rosin (grade M or fourth grade).....	83.5
Chips, needles, etc.....	3.3
Water (by difference).....	1.8
Total.....	100.0

EXAMINATION OF THE VOLATILE OIL.

PHYSICAL PROPERTIES.

The crude oil had a pleasant orangelike odor; specific gravity, 0.6971; and index of refraction, 1.3903. On fractional distillation of 500 grams of the oil the results given in Table 3 were obtained:

TABLE 3.—Fractional distillation of the volatile oil from *Pinus sabiniana*.

Temperature (corrected).	Distillate.		d_{15}° Specific gravity at 15° .	N_{D15}° Index of refraction at 15° .
	Observed.	Cumulative.		
$^\circ\text{C.}$	Per cent.	Per cent.		
96.1-97.3	4.24	4.24	0.6889	1.3890
97.9	7.93	12.17	.6885	1.3898
98.3	9.04	21.21	.6882	1.3898
98.3	8.44	29.65	.6882	1.3898
98.4	8.89	38.54	.6880	1.3898
98.4	8.97	47.51	.6881	1.3898
98.5	8.06	55.57	.6881	1.3898
98.5	7.13	62.70	.6882	1.3898
98.5	6.92	69.62	.6881	1.3898
98.5	6.94	76.56	.6880	1.3899
98.6	6.83	83.39	.6879	1.3899
98.7	6.19	89.58	.6879	1.3899
98.8	5.66	95.24	.6881	1.3900
98.8+	4.66	99.90	.7393	1.4140

IDENTIFICATION OF THE CONSTITUENTS.

Heptane.—It is seen from the data on physical properties given in Table 3 and figure 4 that the crude oil consists of nearly pure heptane, since a single fractionation yields about 90 per cent of an oil with an average boiling point of 98.5° ; specific gravity, 0.6881; and index of refraction, 1.3898; the boiling point has been corrected for barometric pressure using the factor 0.045 as determined from the work of Young ⁽¹⁶⁾ on the vapor pressures of heptane from *Pinus sabiniana*.

Schimmel & Co.⁽⁷⁾ reports that the principal fraction of the oil examined by them boiled between 98.5° and 99° and had a specific gravity of 0.6880 at 15°. Thorpe ⁽¹⁰⁾ obtained the following results on a carefully purified oil: boiling point, 98.42° at 760 mm.; specific gravity, 0.70057 at 0°; refractive index for $D = 1.3879$.

During the distillation of the oleoresin with steam, the last portion of oil coming over had a yellow color and a sharp, penetrating odor. On fractionation of the volatile oil, the fractions had only a faint orange odor, while the residue was colored yellow and had a decidedly higher specific gravity (0.7393), and index of refraction (1.4140), and a strong orange odor. The characteristic odor of the crude oil is apparently due to this yellow oil, which on fractionation becomes concentrated in the residue. Thorpe (10) attributes the odor to a "resenoid matter," while Rabak (3) considers it an ester.

Previous investigators have, in general, found a decidedly higher boiling point for the crude oil. Wenzell (14) gives the boiling point at 101° to 115°, the fractions boiling steadily at 101°. Rabak (3) found it to boil at 100° to 101° and Sadtler (4) at 101° to 103°.

EXAMINATION OF THE ROSIN.

The rosin, grade M, had a specific gravity of 1.073; acid number, 156.7; saponification number, 176.5; and ether number, 19.8; a 5.58 per cent alcoholic solution gave the optical rotation, $a_{D20} = +0.38^\circ$. Rabak (3) states that both the oleoresin and colophony are optically inactive. This statement has not been found to be true of either substance.

All attempts to obtain a crystalline body from the original rosin failed. It seems to be in an amorphous state, noncrystallizable under normal conditions. It was then subjected to distillation in vacuo. Laurent¹ as long ago as 1839 found that the resin acids could be distilled under reduced pressure and the distillate recrystallized. This method has been frequently employed since that time. Rosin is but slightly decomposed under such treatment, and the resin acids recovered are isomers of the formula $C_{20}H_{30}O_2$.

The rosin distilled between 240° and 255° C. at 10 mm. pressure. The viscous distillate solidified on cooling to a hard, transparent, slightly yellow mass, resembling colophony. A portion of the distillate was dissolved in acetone and the solution allowed to stand in a cool place for several days. Large crystals were thus obtained absolutely without color. These were stirred rapidly with small amounts of cold acetone to remove adhering mother liquor, and the solvent was decanted. The crystals were then dissolved in acetone and yielded a colorless solution. Recrystallized twice from the same solvent,

¹ Ann. de Chim. et de Phys., p. 383, vol. 72 (2), 1839.

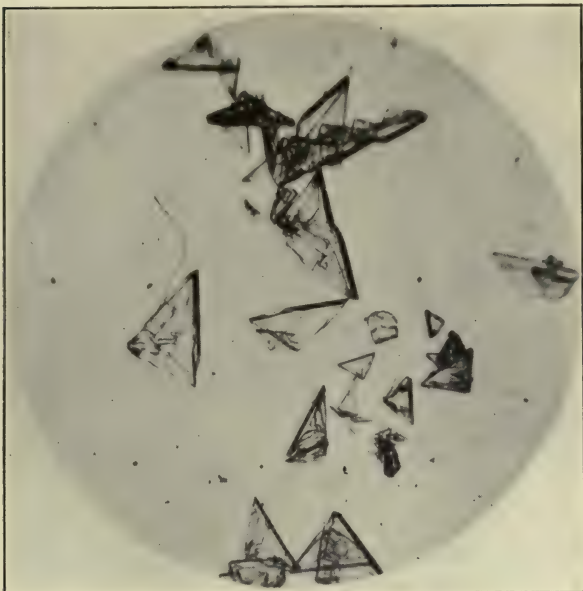


FIG. 1.—RESIN CRYSTALS FROM *PINUS SABINIANA*; FORMED BY RAPIDLY CRYSTALLIZING, FROM ALCOHOL AND HYDROCHLORIC ACID, THE RESIN ACIDS OBTAINED FROM THE ORIGINAL OLEORESIN.

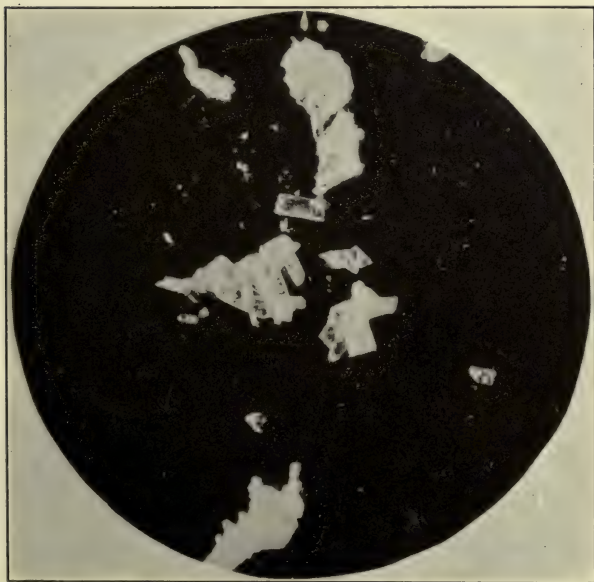


FIG. 2.—RESIN CRYSTALS FROM *PINUS SABINIANA*; FORMED BY CRYSTALLIZING, FROM ACETONE, THE PRODUCT OBTAINED BY DISTILLING THE ROSIN UNDER REDUCED PRESSURE.

the crystals softened at 144° , melted at 151° to 152° , and were completely liquid at 158° . Analysis of the silver salt of the crystals gave:

	Per cent.
0.4689 gram silver salt gave 0.1226 gram of Ag or.....	26. 15
Calculated for Ag ($C_{20}H_{29}O_2$).....	26. 37

The crystals from the crude oleoresin were next examined. A portion of the white crystalline mass which had settled to the bottom of the container was pressed in a hand press, and nearly all the liquid constituents were thus removed. Another method was found to give equally satisfactory results: The crude resinous mass was thrown into a wide Büchner funnel, using silk bolting cloth as a filter; the resin was spread out in a thin layer and suction applied. After two hours a white crystalline mass remained, no longer sticky to the touch, and practically free from volatile oil. After several crystallizations from acetone, finally from methyl alcohol, the crystals melted at 131° ; a higher melting point was not obtained. The alcoholic solution of the crystals was strongly *l*-rotatory. The specific rotation, $[\alpha]_D = -95.82^{\circ}$, was determined from the following values:

$$\begin{array}{ll} a = -3.74^{\circ} & p = 4.764 \\ l = 1 & d = .8193 \end{array}$$

Some of the crystals were dissolved in alcohol and a few drops of concentrated hydrochloric acid added. On cooling, crystals were obtained, in the form of triangular plates, which melted at 158° to 159° . (See Pl. I.) The silver salt of these crystals gave, on analysis, the following results:

	Per cent.
0.2840 gram silver salt gave 0.0751 gram silver.....	=26. 44
Calculated for Ag ($C_{20}H_{29}O_2$).....	=26. 37

CONCLUSIONS.

The volatile oil from *Pinus sabiniana* consists almost entirely of normal heptane. The rosin probably consists of a noncrystallizable form of abietic acid. The rosin distilled under reduced pressure and the crude oleoresin furnished crystalline resin acids of the formula $C_{20}H_{30}O_2$, and it may be assumed that the rosin consists of amorphous acids of the same composition.

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Pinus Lambertiana Dougl.

EXAMINATION OF THE OLEORESIN.

The crude oleoresin from sugar pine (*Pinus lambertiana*) had a specific gravity of 1.0420. The average composition of two analyses follows:

	Per cent.
Volatile oil.....	16.4
Rosin (grade H or seventh grade).....	75.3
Chips, needles, etc.....	3.4
Water (by difference).....	4.9
Total.....	100.0

The oleoresin was stringy, contaminated with needles and fine dust, and foamed badly on distillation with steam; the last portion of volatile oil was removed with great difficulty.

A portion of one sample was heated until it became fluid (about 90° C.) and then filtered. An analysis gave the following results:

	Per cent.
Volatile oil.....	16.2
Rosin (grade K or fifth grade).....	82.0
Dirt.....	0.1
Water (by difference).....	1.7
Total.....	100.0

The filtered material gave a rosin two grades higher than that obtained from the original oleoresin, which well illustrates the effect of trash in darkening the color. From the examination of various

oleoresins it has been found that those of a stringy, viscous, and homogeneous character foam badly on distillation, give up their volatile oil with difficulty, and yield a much darker rosin than is to be expected from "virgin gum." On the other hand, oleoresins, from which the resin acids crystallize and settle out on standing, distill quietly and yield light rosins. The foaming and darkening of the rosin is due partly to the presence of trash, such as needles and chips, and partly to the absorption of oxygen previous to distillation.

EXAMINATION OF THE VOLATILE OIL.

PHYSICAL PROPERTIES.

An oil obtained from oleoresin collected during 1911 had the following properties: Specific gravity, 0.8663; index of refraction, 1.4728; specific rotation, $[\alpha]_D = +10.42^\circ$. Another sample of the oil, amounting approximately to 375 grams, the analysis of which is given in Table 4, had a specific gravity of 0.8658 and an index of refraction of 1.4727.

TABLE 4.—Fractional distillation of the volatile oil of *Pinus lambertiana*.

Temperature (corrected).	Distillate.		d_{15}° Specific gravity at 15°C.	N_{D15}° Index of refraction at 15°C.
	Observed.	Cumulative.		
<i>Degrees, centigrade.</i>	<i>Per cent.</i>	<i>Per cent.</i>		
155.8-157.3	2.98	2.98	0.8531	1.4657
158.6	9.33	12.31	.8620	1.4693
158.9	10.78	23.09	.8629	1.4698
159.3	10.34	33.43	.8632	1.4701
159.7	10.70	44.13	.8633	1.4703
160.3	9.79	53.92	.8633	1.4708
161.3	9.94	63.86	.8634	1.4716
162.5	9.54	73.40	.8633	1.4726
168.4	7.67	81.07	.8624	1.4736
178.8	5.11	86.18	.8548	1.4721
178.8+	13.62	99.80	.9011	1.4912

IDENTIFICATION OF THE CONSTITUENTS.

α -pinene.—The physical properties of the fractions show that the oil consists largely of α -pinene. For further proof a d -rotatory fraction ($\alpha_{D20} = +13.08^\circ$) boiling between 156° and 157° was treated with nitrosyl chloride. The pinene nitrosochloride obtained from this treatment melted at 103° after several precipitations from chloroform with methyl alcohol.

β -pinene.—That portion of the oil boiling between 160° and 168° was oxidized with potassium permanganate to determine the presence of β -pinene. A small amount of nopinic acid, the characteristic oxidation product of β -pinene, was obtained. The acid melted at 125° .

Phellandrene.—By repeated fractionation about 10 c. c. of oil boiling between 169° and 174.5° with a specific gravity of 0.8550 was obtained. When 5 c. c. were treated with bromine, no crystalline derivative was obtained, indicating the absence of limonene; with N_2O_3 a copious crystalline precipitate was obtained, which, when filtered with a force pump, suddenly decomposed into an amorphous mass that could not again be obtained in a crystalline state. It is probable that a small amount of phellandrene was present.

Aliphatic hydrocarbon.—The residue boiling above 178.8° , which amounted to 13.5 per cent, was distilled at 25 mm. pressure—one-fourth distilled between 110° and 130° and the remainder between 130° and 145° . The portion boiling between 110° and 130° at 25 mm. was split up into four fractions whose specific gravities and indices of refraction showed that it could not consist of terpenes. These fractions were accordingly combined and treated with concentrated sulphuric acid until no further reaction was caused by the addition of more acid. The residual oil boiled between 194° and 201° C. at 742.7 mm.; the specific gravity was 0.7549 and the index of refraction, 1.4249. The decrease in the index of refraction of the tenth fraction, shown in Table 4, must be due to this oil, since all terpenes of as high a boiling point have a much higher index of refraction. From its physical and chemical properties this oil may be a hydrocarbon of the paraffin series. Owing to the small amount of material available for analysis no more definite information could be obtained. The results are based on analysis of material collected during the years 1910 and 1911.

Aromadendrene.—The portion of the residue boiling between 130° and 145° at 25 mm. pressure consisted of a sesquiterpene. It did not yield a crystalline derivative with Br, N_2O_3 , or HCl. The carefully purified oil had the following properties: Boiling point, 144° to 148° at 30 mm. (250° to 255° at 739.9 mm.); specific gravity, 0.9238; index of refraction, 1.5006; specific rotation, $[\alpha]_D = +37.88^{\circ}$ determined from a 33.33 per cent ethereal solution. When a few drops were dissolved in glacial acetic acid in a test tube, and bromine vapors were allowed to flow into the tube, a series of beautiful color reactions were obtained. The solution first turned crimson, then violet, and on standing 5 to 10 minutes became a deep blue. In its physical properties and color reactions the sesquiterpene from *Pinus lambertiana* agrees well with the sesquiterpene "aromadendrene," described by Smith¹ as occurring in Australian eucalyptus oils. Aromadendrene shows the color reactions given above, boils between 260° and 265° C. at atmospheric pressure, and has a specific gravity of 0.9229 to 0.9249 at 19° C.

¹ Jour. and Proc. Roy. Soc. N. S. W., pp. 124-126, vol. 25, 1901.

EXAMINATION OF THE ROSIN.

A sample of H rosin had a specific gravity of 1.078; acid number, 142.7; saponification number, 152.6; ether number, 9.9. The rosin distilled at 250° to 275° at 30 mm. pressure. The viscous distillate hardened to a light yellow, amorphous mass on cooling. Attempts to secure a crystalline resin acid from the original rosin and its distillation product were unsuccessful.

CONCLUSIONS.

From the distillation data it has been estimated that the volatile oil of the sugar pine consists of 70 to 75 per cent *d*- α -pinene; about 5 per cent β -pinene; 2 to 3 per cent of a terpene, probably phellandrene; 2 to 3 per cent of what appears to be an aliphatic hydrocarbon; and 10 to 12 per cent of a sesquiterpene which agrees well in its properties with those of "aromadendrene." The portions of the oil boiling between 170° to 210° C. should be investigated further.

Pinus Contorta Loud.

LITERATURE.

Blasdale¹ examined the oil obtained from oleoresin scraped from peeled lodgepole pines (*Pinus murrayana*).² He states that the major portion of the oil distilled between 158° and 160°, but portions boiled as high as 180°. The fraction boiling between 158° and 160° had a specific gravity of 0.8640; an index of refraction of 1.4765; and specific rotation, $[\alpha]_D = -15^\circ 23'$. Without further examination the oil is stated to "consist of terpenes."

EXAMINATION OF THE OLEORESIN.

The crude material which was examined by the author had a specific gravity of 1.0210 and gave on analysis the following percentages:

	Per cent.
Volatile oil.....	14.7
Rosin (grade I or sixth grade).....	77.7
Chips, needles, etc.....	2.5
Water (by difference).....	5.1
Total.....	100.0

The oleoresin was very dark, stringy, and badly contaminated with needles, chips, and fine dirt. It foamed excessively on distillation with steam, and the last traces of oil were removed only with difficulty.

¹ Jour. Am. Chem. Soc., pp. 163-164, vol. 23, 1901.

² A distinction is no longer maintained between *Pinus contorta* and *Pinus murrayana*.

EXAMINATION OF THE VOLATILE OIL.

PHYSICAL PROPERTIES.

The original oil from the oleoresin collected during the summer of 1911 had the following properties: Specific gravity, 0.8518; index of refraction, 1.4862; specific rotation, $[a]_D = -20.12^\circ$. The oil examined from oleoresin collected in 1910 had: Specific gravity, 0.8549; index of refraction, 1.4860. About 400 grams of the latter oil were submitted to fractional distillation with the results shown in Table 5.

TABLE 5.—*Fractional distillation of the volatile oil from Pinus contorta.*

Temperature (corrected).	Distillate.		d_{15}° Specific gravity at 15°.	$N_{D_{15}}^\circ$ Index of refraction at 15°.
	Observed.	Cumulative.		
° Centigrade.	Per cent.	Per cent.		
164.4–170.6	2.89	2.89	0.8547	1.4793
171.6	8.55	11.44	.8546	1.4814
172.6	9.08	20.52	.8542	1.4823
173.6	10.77	31.29	.8534	1.4835
175.3	10.22	41.51	.8522	1.4852
177.2	9.79	51.30	.8514	1.4861
177.6	10.03	61.33	.8507	1.4871
177.9	10.23	71.56	.8502	1.4880
178.4	9.26	80.82	.8498	1.4891
178.9	4.15	84.97	.8498	1.4894
178.9+	14.83	99.80		

The oil had a very characteristic odor; it was pungent when freshly distilled and, after standing some time, was orange-like. The oil polymerized very considerably at its boiling point. The 15 per cent residue remaining in the distilling flask solidified to a solid, amber-colored mass resembling colophony, insoluble in alcohol, but soluble in ether and chloroform. To avoid loss of oil by polymerization it must be distilled under reduced pressure. The oil carefully purified and finally distilled over metallic sodium, had the following properties: Boiling point, 60° at 11 mm.; specific gravity, $\frac{21^\circ}{15^\circ}$ 0.8460; refractive index, 1.4861; specific rotation, $[a]_D = -12.36^\circ$.

IDENTIFICATION OF CONSTITUENTS.

α -pinene.—The first fraction was redistilled over metallic sodium and tested for α -pinene by treatment with ethyl nitrite and hydrochloric acid. No crystalline nitrosochloride or blue coloration was obtained; however, the failure to secure a precipitate is not conclusive proof of the absence of α -pinene,¹ especially when the oil is highly active.²

Phellandrene.—The ease with which the oil polymerized, and its physical properties, pointed to phellandrene. When the original oil was treated with potassium nitrite and acetic acid, or when any

¹ Wallach: *Annalen*, p. 6, vol. 368, 1909.² Tilden: *Jour. Chem. Soc.*, p. 759, vol. 85, 1904; also Kremers: *Pharm. Rundschau*, p. 135, vol. 13, 1895.

of its fractions were so treated, a copious precipitate of phellandrene nitrite was obtained. After repeatedly dissolving in chloroform and precipitating with methyl alcohol the nitrite melted at 90° when slowly heated, and at 103° when heated rapidly.

From the data obtained on fractionation it was thought that perhaps both α - and β -phellandrene were present. Wallach¹ states: " β -phellandrene appears to possess a somewhat lower boiling point and higher specific gravity than α -phellandrene." He shows, also, the existence of an α - and β -nitrite of α -phellandrene, and an α - and β -nitrite of β -phellandrene melting at 112° to 113° or 113° to 114° , 105° , 102° , and 97° to 98° ,² respectively. These melting points were obtained by Wallach, through rapid heating. Although the data in Table 5 and the curves of figures 1 and 2 indicate strongly that both forms of phellandrene are present, in no case was it possible to obtain a nitrite of lower, or higher, melting point from any of the fractions by solution of the nitrite in acetone, and fractional precipitation with water, if the nitrite was purified until perfectly white. Examination for limonene also gave negative results. The oil must, therefore, consist largely of β -phellandrene. So far as known, this terpene has never before been detected in the oil from the oleoresin of any member of the genus *Pinus*.

EXAMINATION OF THE ROSIN.

The rosin was not completely soluble in 95 per cent alcohol. A white curdy residue was obtained, which indicated the presence of some of the oil that had been polymerized during distillation. In appearance it is exactly like the polymerized residue from the distilled oil when digested with alcohol. The oil accordingly polymerizes with great readiness, for the distillation was conducted with steam, and the temperature of 145° C. was not exceeded in drying the rosin.

The rosin, grade I, had the following properties: Specific gravity, 1.061; acid number, 131.2; saponification number, 139.6; ether number, 8.4. About 80 per cent of crude crystals in the form of triangular plates, and melting at 155° , were obtained by crystallization from alcohol, diluted with concentrated HCl. After several crystallizations from acetone the abietic acid melted at 159° to 160° .

For further confirmation, the silver salt of the abietic acid was obtained and analyzed with the following results:

	Per cent.
0.2340 gram silver salt gave 0.0611 gram silver.....	=26.11
.2470 gram silver salt gave .0647 gram silver.....	=26.19
Mean.....	=26.15

Silver abietate, $\text{Ag}(\text{C}_{20}\text{H}_{20}\text{O}_2)$, requires 26.37 per cent silver.

¹ Annalen, pp. 42-43, vol. 336, 1904.

² Ibid., pp. 15 and 44.

CONCLUSIONS.

The volatile oil of *Pinus contorta* consists almost entirely of *l*- β -phellandrene. The colophony contains about 80 per cent abietic acid.

Pinus edulis Engelm.

EXAMINATION OF THE OLEORESIN.

The oleoresin of piñon pine (*Pinus edulis*) contains very large resin acid crystals and has a characteristic odor resembling *thuja* leaf oil. It has a specific gravity of 1.0238.

The average composition as shown by the analysis of five consecutive dippings is:

	Percent.
Volatile oil.....	20.0
Rosin (grade I to WG).....	76.5
Trash.....	0.9
Water (by difference).....	2.6
Total.....	100.0

EXAMINATION OF THE VOLATILE OIL.

PHYSICAL PROPERTIES.

The oil had the characteristic odor of the oleoresin, due to the presence of a sesquiterpene; the specific gravity was 0.8680; the index of refraction, 1.4707; and the specific rotation $[\alpha]_D = +19.26^\circ$. On fractional distillation the results shown in table 6 were obtained:

TABLE 6.—Fractional distillation of the volatile oil of *Pinus edulis*

Temperature (corrected).	Distillate.		d_{15}° Specific gravity at 15° C.	$N_{D_{15}^\circ}$ Index of refraction at 15° C.
	Observed.	Cumulative.		
<i>Degrees Centigrade.</i>	<i>Per cent.</i>	<i>Per cent.</i>		
155.9-157.4	5.10	5.10	0.8617	1.4677
157.6	10.66	15.76	.8617	1.4683
157.9	10.28	26.04	.8621	1.4687
158.4	8.99	35.03	.8619	1.4689
158.9	10.54	45.57	.8616	1.4689
160.0	11.04	56.61	.8611	1.4693
162.4	8.76	65.37	.8609	1.4696
176.5	9.72	75.09	.8605	1.4713
194.9	3.87	78.96	.8616	1.4723
194.9+	20.74	99.70	.9129	1.4837

IDENTIFICATION OF CONSTITUENTS.

α -pinene.—The results obtained on fractional distillation show that the oil consists largely of *α -pinene*. For further confirmation the d-rotatory first fraction was treated with nitrosyl chloride; the pinene nitrosochloride obtained melted at 103° after purification.

Table 6 shows that the specific gravity of the fractions gradually decreases from the third to the ninth fraction. This may be due to the presence of a small amount of limonene. However, on redistillation, about 90 per cent of the fractions which first boiled between 162.4° and 194.9° , boiled between 156° and 165° , which showed that they consist largely of pinene. The higher boiling point on the first distillation may be explained by the retarding effect of the large amount of high boiling sesquiterpene present.

β -pinene.—The portion which boiled between 160° and 170° on redistillation, amounting to 12 per cent, was oxidized with $K_2Mn_2O_8$. About 0.5 per cent of nopinic acid, the characteristic oxidation product of β -pinene, was recovered; it melted at 123° .

Cadinene.—The residue boiling above 194.9° distilled between 118° and 145° at 20 mm. pressure. A fraction representing 50 per cent of the total was collected between 135° and 140° , the specific gravity of which was 0.9173, the index of refraction 1.4925 and the specific rotation $[\alpha]_D = +15.41^{\circ}$; the color was faint green. This fraction, to which the characteristic odor of the original oil is due, gave a dihydrochloride melting at 118° . A 5.94 per cent solution of the dihydrochloride in ether gave the optical rotation $\alpha_{D20} = -1.89^{\circ}$. Cadinene is accordingly the sesquiterpene present. When five drops of the cadinene fraction were dissolved in 2 c. c. of acetic anhydride and a drop of concentrated sulphuric acid added a green color was produced, which became blue, then purple, and finally red. When glacial acetic acid¹ is used in place of acetic anhydride a red color is produced almost immediately. This appears to be the first recorded occurrence of cadinene in a turpentine oil.

EXAMINATION OF THE ROSIN.

A sample of WG rosin had the following properties: Specific gravity, 1.060; acid number, 155.2; saponification number, 164.1; ether number, 8.9. The rosin distilled between 240° to 255° at 10 mm. pressure. A few long prismatic crystals formed on the sides of the receiver, but could not be isolated because of adherent oily products. The viscous distillate hardened to a faint yellow, amorphous mass. All attempts to secure a crystalline body from either the original rosin or the distilled rosin were unsuccessful.

A portion of the white resin acids from the original oleoresin, which had settled to the bottom of the container, was pressed in a hand press until the greater portion of the intermingled liquid products had been removed. The white, waxy cake, after four crystallizations from acetone, yielded crystals melting at 129° to 130° . When these were dissolved in hot absolute alcohol, and a few drops

¹ Compare Wallach, *Annalen*, p. 87, vol. 238, 1887.

of concentrated hydrochloric acid were added, microscopic triangular plates separated on cooling. These plates melted at 137° and were completely liquid at 148° . The activity of the resin acid was determined in an alcoholic solution, the specific rotation, $[\alpha]_D = -52.77^{\circ}$, being computed from the following values:

$$\begin{array}{ll} a = -0.57^{\circ} & p = 1.33 \\ l = 1 & d = 0.8122 \end{array}$$

Analysis of the silver salt of the resin acid gave the following results:

0.1867 gram silver salt gave 0.0494 gram silver.....	Per cent. =26.46
Calculated for $\text{Ag}(\text{C}_{20}\text{H}_{30}\text{O}_2)$	=26.37

CONCLUSIONS.

From the distillation data it has been estimated that the volatile oil from the oleoresin of *Pinus edulis* contains approximately 70 to 75 per cent α -pinene, about 5 per cent β -pinene, and 15 to 20 per cent d -cadinene. The oleoresin yields a resin acid of the formula $\text{C}_{20}\text{H}_{30}\text{O}_2$; although no crystalline resin acid was obtained from the colophony it may be assumed to consist of amorphous acids of the preceding formula.

SUMMARY.

With the exception of the digger pine, the volatile oil from all the species examined consists of terpenes ($\text{C}_{10}\text{H}_{16}$), and sesquiterpenes ($\text{C}_{15}\text{H}_{24}$). The oil from the digger pine consists of the hydrocarbon heptane (C_7H_{16}), a normal constituent of petroleum.

COMPARISONS WITH COMMERCIAL NAVAL STORES.

VOLATILE OIL.

In Table 7 and in figures 1 to 4 the various oils examined are compared with an average gum turpentine which consists of a mixture of the volatile oils of the Cuban pine (*Pinus heterophylla* Ell.) and longleaf pine (*Pinus palustris* Mill.):

TABLE 7.—Characteristics of the volatile oil from the oleoresin of various pines.

Source of oil.	$n_{D_{15}}^{\circ}$ Index of refraction at 15°C .	d_{15}° Specific gravity at 15°C .	Boiling points of first 90 per cent of the distillate.	Principal constituent.
Western yellow pine, California.....	1.4772	0.8625	164.0 to 177.5°C	β -pinene.
Western yellow pine, Arizona.....	1.4727	.8639	157.6 to 175.0	α -pinene.
Digger pine, California.....	1.3903	.6971	96.2 to 98.8	Heptane.
Sugar pine, California.....	1.4727	.8658	155.8 to 180.0	α -pinene
Lodgepole pine, California.....	1.4860	.8549	164.4 to 179.0	β -phellandrene.
Piñon pine, Colorado.....	1.4707	.8680	(1)	α -pinene.
Gum turpentine ²	1.4730	.8685	156 to 165	α -pinene.

¹ The first 80 per cent distills between 156° and 200°C . ² From Cuban and longleaf pines.

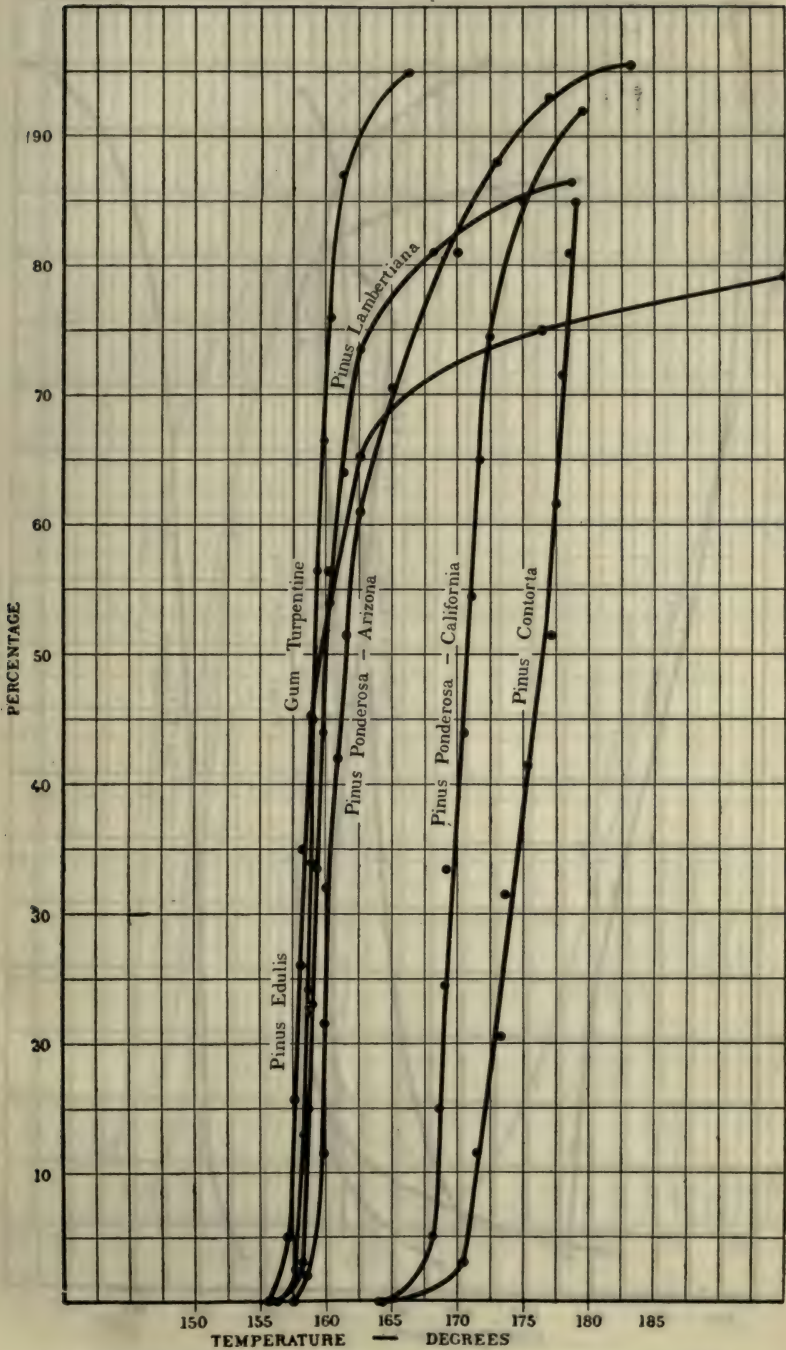


FIG. 1.—Boiling-point curves for the volatile oil obtained from the oleoresin of various pines.

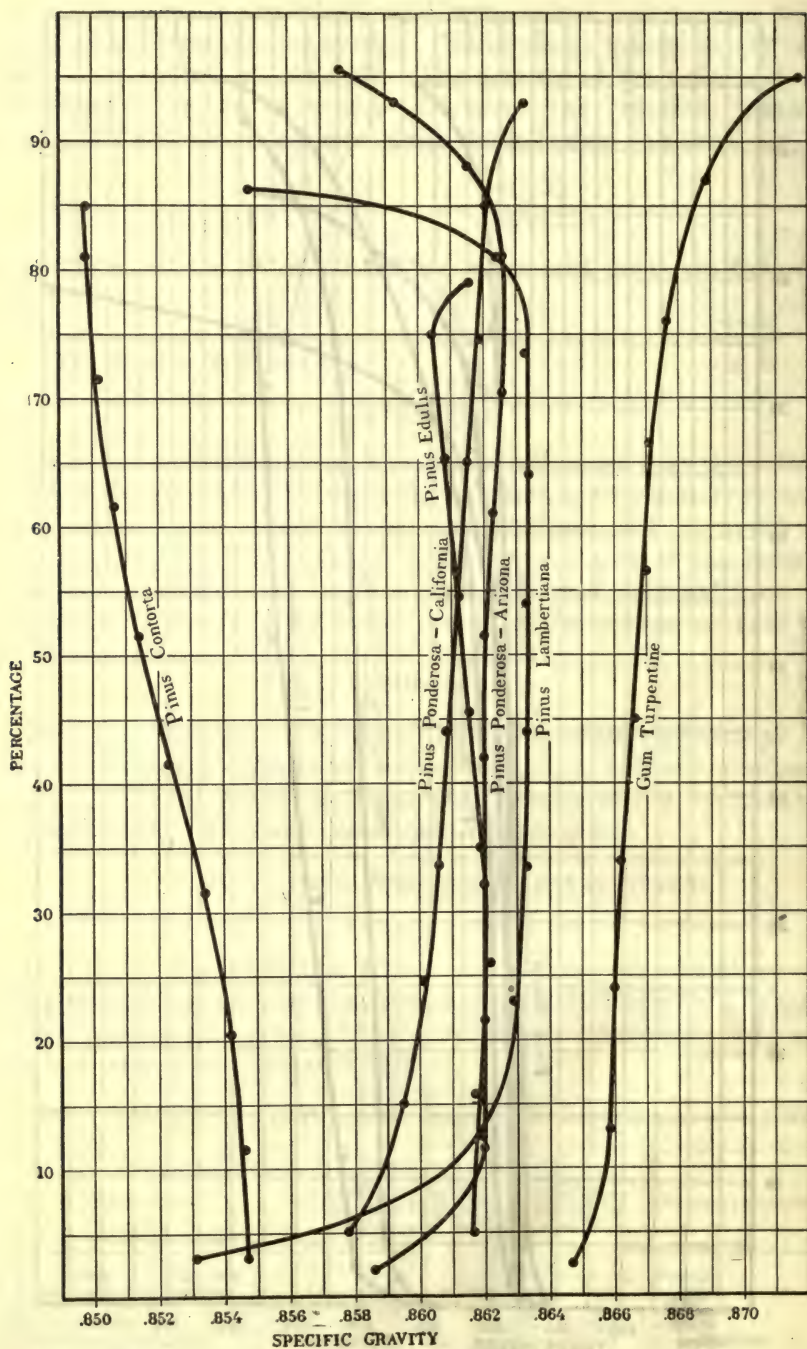


FIG. 2.—Specific-gravity curves for the volatile oil obtained from the oleoresin of various pines.

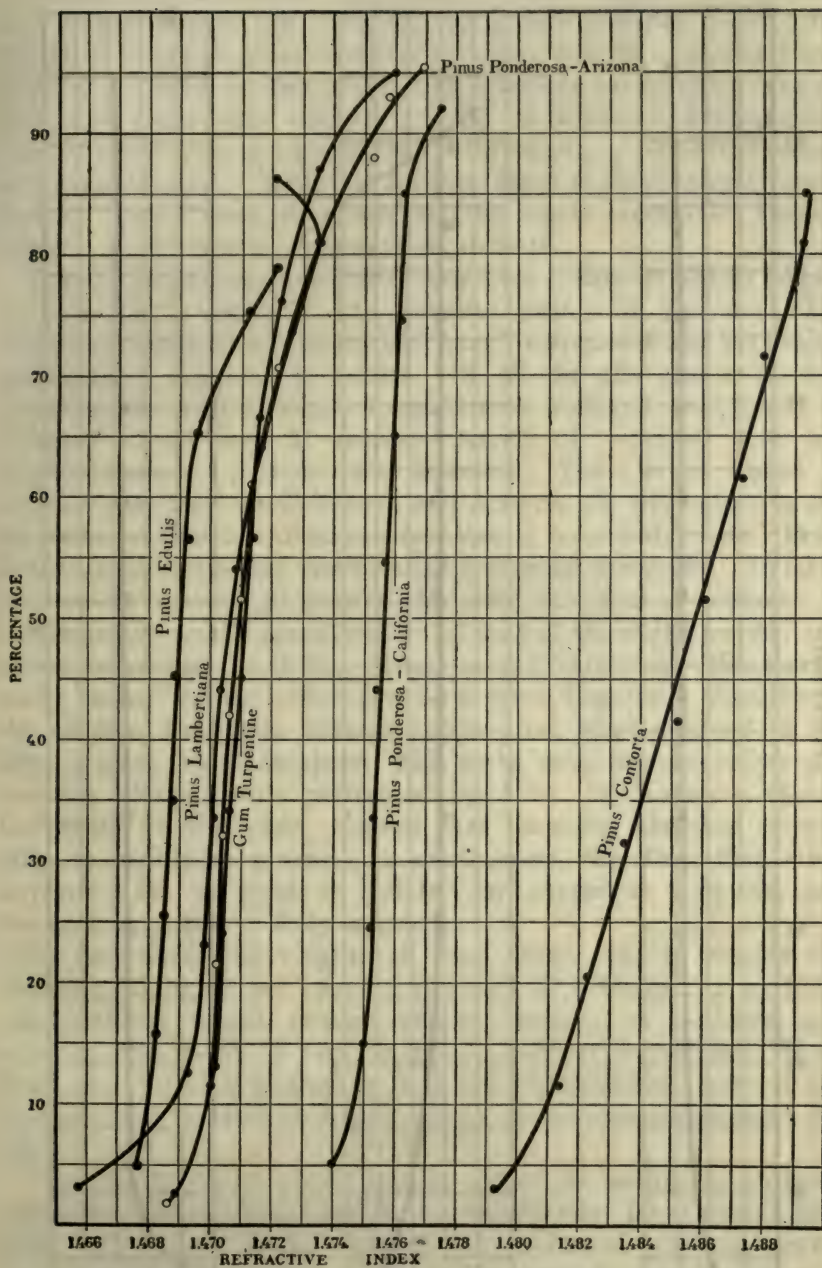


FIG. 3.—Index of refraction curves for the volatile oil obtained from the oleoresin of various pines.

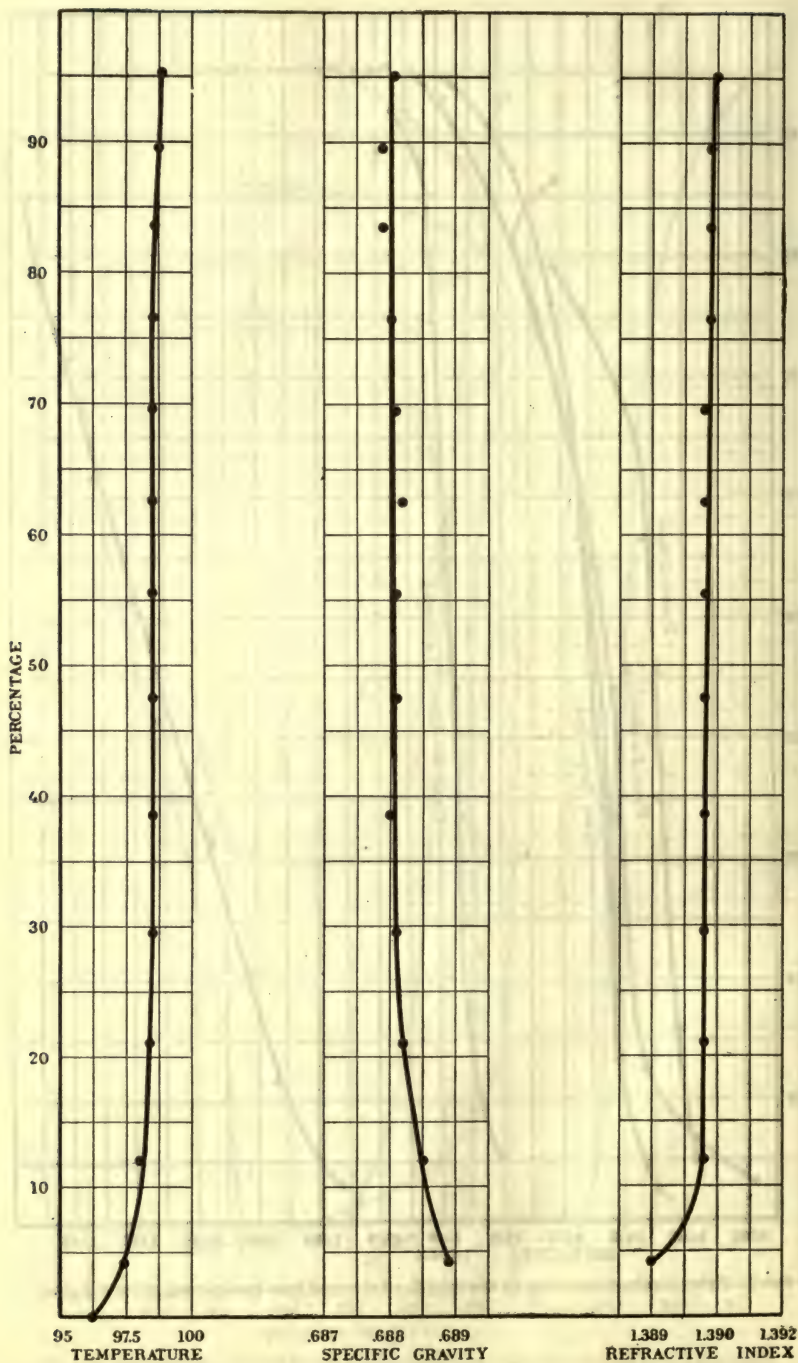


FIG. 4.—Boiling point, specific gravity, and index of refraction curves for the volatile oil from the oleoresin of *Pinus sabiniana*.

French and American turpentine consist largely of α -pinene and 95 per cent of the fresh oils will generally distill below 170° . The oil of lodgepole pine consists of the unstable terpene phellandrene, and has a higher boiling point than ordinary turpentine. The oil from the sugar pine consists mainly of α -pinene, but contains about 25 per cent of high-boiling constituents, a portion of which is a sesquiterpene. However, because there is an extremely small flow of "gum" from the sugar and lodgepole pines their volatile oils will hardly become commercial products.

The oil from the western yellow pine from California distills largely between 168° and 172° and consists mainly of β -pinene. The latter is a normal constituent of "gum" turpentine and invariably accompanies α -pinene in nature. Of all the oils examined, that from the western yellow pine from Arizona conforms most nearly to ordinary turpentine. It consists largely of α -pinene with but small amounts of β -pinene and limonene. There is no reason to suppose that both the California and Arizona oils will not serve the purposes for which ordinary turpentine is commonly used. Practical tests are necessary, however, to determine this point. In addition to its solvent properties the chief function of α -pinene in turpentine is usually considered to be that of absorbing oxygen and hence promoting the drying of paints and varnishes. Whether it really has a "drying" effect has been often disputed.¹ Samples of the oil from California western yellow pine, when exposed to the light in glass vessels stoppered with corks, soon became yellow and resinous. Experiments performed by Prof. E. Kremers, of the University of Wisconsin, showed that limonene absorbed oxygen fully as rapidly as pinene. It would seem, therefore, that other terpenes have as great an affinity for oxygen as α -pinene, and their solvent power is fully as great.

By redistilling the volatile oil from piñon pine to remove the cadinene 75 to 80 per cent of oil could be collected up to 175° ; this distillate would consist almost entirely of α -pinene and β -pinene, and would be very similar to ordinary turpentine. However, the distillate retains in part the characteristic odor of the cadinene residue and this odor might prove objectionable to the user.

The volatile oil of digger pine is not at all similar to ordinary turpentine oils and consequently can not be used for the same purpose. It consists almost entirely of heptane of uniform specific gravity and boiling point. It is readily purified by agitation with a small amount of concentrated sulphuric acid. Various industries should find in this a cheap and valuable solvent where uniformity is required.

¹ Compare Powney, "The influence of solvents on the drying of linseed oil," *Analyst*, pp. 192-198, vol. 35, 1910.

ROSIN.

Table 8 contains the constants of the various "rosins" as well as the constants of a sample of commercial rosin from the Southern States which represents a mixture of colophony from the longleaf and Cuban pines:

TABLE 8.—*Chemical and physical constants of rosins from various species.*

Source of "rosin."	Grade.	d_{15}° Specific gravity at 15° C.	Acid number.	Saponifica- tion number.	Ether number.
Western yellow pine, California.....	WW	1.068	148.8	161.3	12.5
Digger pine.....	M	1.073	156.7	176.5	19.8
Sugar pine.....	H	1.078	142.7	152.6	9.9
Lodgepole pine.....	I	1.061	131.2	139.6	8.4
Pinon pine.....	WG	1.060	155.2	164.1	8.9
Cuban and longleaf pine mixed.....	WW		161.5	170.4	8.9

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Contribution from the Forest Service, Henry S. Graves, Forester.

September 10, 1914.

Division of Forestry

University of California

(PROFESSIONAL PAPER.)

YIELDS FROM THE DESTRUCTIVE DISTILLATION OF CERTAIN HARDWOODS.¹

By L. F. HAWLEY and R. C. PALMER, *Chemists in Forest Products.*

PURPOSE OF EXPERIMENTS.

The chief hardwoods used in this country for distillation are beech, birch, and maple. Only mill and forest waste and trees unmerchantable for lumber are now ordinarily used, although some material suitable for lumber still finds its way to the distillation plants. Such southern hardwoods as the oaks, red gum, tupelo, and hickory have not been important in distillation, and no information has existed in regard to the amount of the various products which could be obtained from them. Nor has information been available on the relative value of the commonly used species, or of the different forms of material, such as body wood, limbs, and slabs. The investigation here described was undertaken in order to supply this information and to aid in this way the utilization of materials now wasted.

METHOD OF INVESTIGATION.

GENERAL PLAN.

Since conditions of distillation influence the yield of products, results obtained in the laboratory could not be compared directly with those obtained in commercial operations. In order to have a direct comparison between the species commonly used and the ones which are not, it was therefore necessary to include both classes in the investigation.

The various materials were distilled under similar conditions, and their products analyzed by the same methods. In order to avoid errors due to differences in yields from different trees of the same species, in most cases an average sample of material from two or

NOTE.—Gives results of experiments in destructive distillation of hardwoods. Of interest to manufacturers of by-products.

¹ The investigation the results of which are given in this bulletin were conducted at the Forest Products Laboratory, Madison, Wis., maintained in cooperation with the University of Wisconsin.

three trees was distilled. Further, the average yields from the heartwood¹ of several trees were in a few instances compared with the yields from lumber of the same species. Differences in yields may also occur in trees of the same species grown in different localities, and for this reason the results obtained are averaged separately when more than one locality is represented. At least two distillations were made of each kind or form of material tested.

Different forms of wood—such as body wood (wood free from bark), slabs, limbs, etc.—were distilled, but the proportion of each used in commercial practice varies with different plants and localities, so that it is not possible to assume a proportion representing average conditions. For this reason the yields from different forms of wood of the same species are presented separately. The corresponding yield per given weight of wood, made up of any proportion of the various forms, can readily be calculated. However, as a basis of comparison between the species, the average yields from all heartwood runs (including lumber) are taken arbitrarily as the species value. The mean of the heartwood and slab-wood yields is also given wherever both forms were distilled.

THE RETORT.

Figure 1 shows the construction of the retort in which the distillations were made. The retort proper *A* was surrounded by the oil jacket *B*, which was filled with a high-flash-test cylinder oil. The outlet pipe *C* connected the retort with an ordinary worm condenser (not shown). The pyrometer tubes, 1, 2, 3, 4, and 5, made it possible to measure the temperature at various places within the retort. The retort was mounted on an iron stand, was insulated on all sides, and heated by a row of gas burners underneath. The flames from the burners played chiefly up one side of the cylinder and induced a fairly good circulation of the hot oil around the retort.

PREPARATION OF MATERIAL.

The forms of material used varied to some extent with different species, but most of them consisted of round bolts. These were sawed into slabs and heartwood in about the same proportion as would occur in ordinary sawmill practice, and the percentage of bark on the slabs was roughly determined. Sticks were prepared from 6 to 8 square inches in cross section and a trifle less than 18 inches long. Just before each charge of wood was weighed, six 1-inch sections, each cut from a different stick and in each case from a different part of the stick, were taken for moisture determinations.

In the comparative distillations on bark and sapwood the material was taken from the same bolts. When limbs were used they were

¹ The term "heartwood" as used in this paper applies to the material left after the slabs have been removed from a bolt or log. It was in all cases entirely free from bark, but small amounts of sapwood sometimes remained. Lumber is considered as made from heartwood as thus defined.

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YIELDS FROM DISTILLATION OF CERTAIN HARDWOODS

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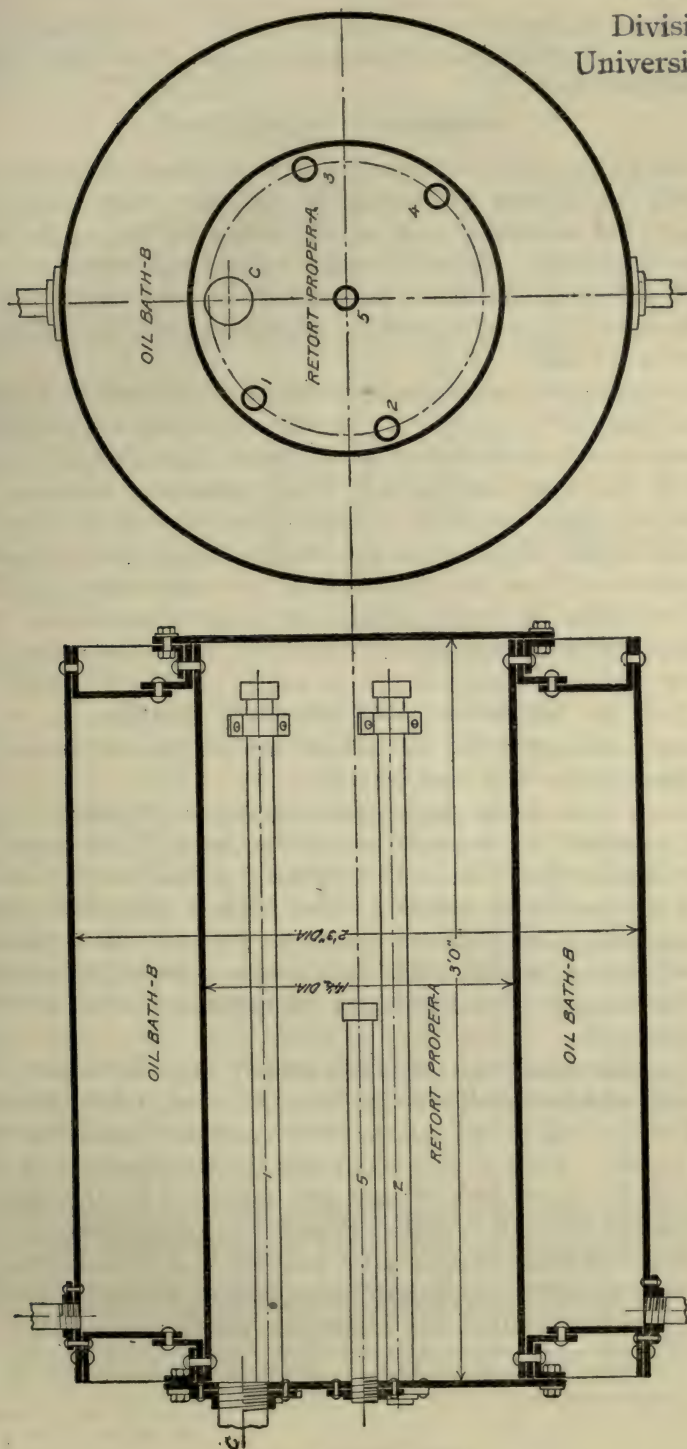


FIG. 1.—Experimental retort.

taken from the same trees as the body wood. In the case of factory waste or lumber there was, of course, no record of the trees from which the material came.

TEMPERATURES OF DISTILLATION.

It was found that the temperatures in pyrometer tubes 1, 2, 3, and 4, which are all near the surface of the retort, were always within 15° to 20° of each other, and usually within 10° during the last part of the distillation. Tube 1 was the hottest and tube 5 the coolest. It was, therefore, considered unnecessary to take temperature readings in tubes 2, 3, and 4, and the records contain the readings from tubes 1 and 5 only.

The maximum temperatures obtained in the various distillations ranged from 327° C. to 415° C., and the maximum temperatures near the surface and at the center of the retort often differed as much as 60° C. in the same distillation. These differences, however, did not appreciably affect the yields of alcohol and acetic acid, since in some instances higher temperatures gave higher yields, and in others lower. It is also found that the charcoal from low-temperature distillations, when redistilled in small samples at temperatures above 400° C., produced only small amounts of acetic acid (equivalent to an increase of 2 per cent of the original yield of acid). It was considered, therefore, that the distillations were practically complete, as to alcohol and acetic acid, provided all parts of the charge had been subjected to a temperature of at least 320° C.¹

In most of the distillations, on account of the exothermic character of the reaction, the temperature at the center of the retort finally became higher than that at the surface. It was the heat developed during the exothermic reaction which made it difficult to obtain the same maximum temperatures in all distillations; after the reaction was well started at the surface its progress toward the center was spontaneous and the maximum temperature could not be fully controlled.

The maximum temperature was usually kept below 260° C. until the water was nearly all expelled from the wood and the temperature at the center had risen to about 190° C., when it was allowed to rise more rapidly. Only in this way could the temperatures at different points in the retort be kept near one another. By this means also the possible effect of variation in moisture content was minimized, since the slow preliminary heating resulted in a partial drying of the wood, and the different charges had therefore nearly the same moisture content at the time the destructive distillation began.

¹ See Klason, von Heidenstam and Norlin, *Arkiv for Kemi Min. och Geol.* 1908, III, 9.

TABLE 1.—*Sample data sheet.*

Shipment No. 197.

Sample Nos. 21 and 22.

January 14, 1913.

Actual weight of charge 69.86 pounds.

Dry weight of charge 63.10 pounds.

Project No. 152.

Run No. 97.

Birch slab wood.

10 per cent to 14 per cent bark.

Time.	Temperature.		Total distillate.	Remarks.
	Tube No. 1.	Tube No. 5.		
<i>January 13.</i>	° C.	° C.	c. c.	
4.30 p. m.		20		Retort charged; gas on one-half.
<i>January 14.</i>				
8.20 a. m.	234	180	3,200	Gas on full.
10 a. m.	280	220	4,200	
10.50 a. m.	303	246	5,700	
11.06 a. m.	312	263	6,700	
11.16 a. m.	317	278	7,700	
11.22 a. m.	322	291	8,700	
11.27 a. m.	329	305	9,700	
11.33 a. m.	336	318	10,700	Gas off.
11.40 a. m.	342	325	11,700	
11.51 a. m.	342	351	12,700	
12 a. m.	344	370	13,200	
12.06 a. m.	341	378	13,500	
12.11 a. m.	339	379		Maximum temperature.
12.15 a. m.	337	376		
1.50 a. m.	282	284	14,000	
<i>January 15.</i>				
8 a. m.		50	14,200	

Total distillate=32.83 pounds.

COLLECTION AND ANALYSIS OF PRODUCTS.

A typical data sheet is shown in Table 1. The time and temperatures were read as every liter or half liter of distillate was collected. In a few distillations separate titrations for acetic acid were made on the first several fractions of one-half liter or one liter each, but in general all the distillate was mixed for analysis.¹ The distillate was allowed to settle for at least 24 hours. At the end of that time the tar and pyroligneous acid were separated by decantation, and the volume and weight of each determined. The charcoal was allowed to cool in the retort over night, and was weighed after separation from the "tar coke." Tar coke refers to the material occurring in the retort that was clearly a residue from the distillation of tar. This was weighed separately. The gas was computed by difference, and no determination of its composition was made.

PYROLIGNEOUS ACID.

The pyroligneous acid was analyzed by the methods described by Klar² for the determination of acetic acid, wood alcohol, and dis-

¹ The acetic acid in that part of the distillate (consisting usually of water) which came over before true destructive distillation began amounted to from 8 to 10 per cent of the total acetic acid; the alcohol in the same part amounted to about 1 per cent of the total alcohol. The volatile acids obtained at temperatures below the point at which the wood begins to distill destructively, say 280° C., must have an origin different from that of the acid obtained during the destructive distillation. It is probably formed by action of the water on the wood fiber at high temperatures similarly to the acid obtained by hydrolysis as reported by Cross (Dissertation, Göttingen, 1910).

² Technologie der Holzverkohlung, p. 337.

solved tar. For the acetic-acid and dissolved-tar determinations 100 c. c. of pyroligneous acid were distilled at a maximum temperature of 140° C. until practically no further distillate appeared, when 50 c. c. of water were added and distilled off as before. The residue in the flask was weighed and computed as dissolved tar, while for the acetic-acid determination an aliquot part of the distillate was titrated with normal sodium hydroxide solution, with phenolphthalein as indicator.

The wood alcohol was determined by distilling 60 per cent from a 1-liter sample of the pyroligneous acid and adding an excess of sodium hydroxide to the distillate, again distilling 60 per cent, and after again adding sodium hydroxide, making a third distillation of 60 per cent. The final distillate was accurately weighed, and the specific gravity determined by means of a Westphal balance at room temperature and corrected to 15° C. by using the tables of Dittmar and Fawsitt.¹ In correcting the specific gravity for temperature it is necessary to consider both the concentration of alcohol and the range of temperature.

TAR.

The amount of acetic acid in the settled tar was determined, after Klar, by distilling 100 grams of the tar at 130° to 140° until the watery distillate ceased, then passing steam through the residue until no more acid was found in the distillate, the latter being then titrated, as in the pyroligneous-acid analysis, and added to that found in the pyroligneous acid.

COMPUTATION OF RESULTS.

All the yields of products were first computed to a percentage of the dry weight of the material distilled, since only on this basis are the results directly comparable, the effect of varying percentages of moisture in air-dry wood and of differences due to weight per unit volume being eliminated. But because the unit of measurement for wood is the cord, and the capacity of a plant is naturally expressed in this unit, a comparison between the various species is made also on the cord basis. A cord was assumed to contain 90 cubic feet of actual wood, and its weight was derived from the average weight per cubic foot of air-dry wood of different species as given by Snow.²

The actual volume of a cord differs, of course, for different forms of material, due to variation in diameter and shape among the individual pieces. Also, differences in density exist between wood from different parts of the tree and between wood and bark; hence between forms of material containing different proportions of wood and bark. For these reasons it was impossible to estimate closely the weight per cord of the several forms as compared with each other, and the

¹ Trans. Roy. Soc. Edin., vol. 33. Quoted in Smithsonian Physical Tables.

² The Principal Species of Wood, by C. H. Snow.

weight is therefore assumed to be the same for all forms, and the yields per cord computed on this basis.

For comparison with commercial conditions it is better also to express the yields per cord in terms of commercial products, and so they are computed as 82 per cent crude wood alcohol and 80 per cent gray acetate of lime.

YIELDS ON PERCENTAGE WEIGHT BASIS.

ALCOHOL AND ACETIC ACID.

VARIATION AMONG SPECIES.

The average yields of total acetic acid and wood alcohol, expressed in percentages of the oven-dry weight of the material distilled, are shown in Table 2.

TABLE 2.—Yields of alcohol and acetic acid in percentages of oven-dry weight of material distilled.

YIELD OF WOOD ALCOHOL (100 PER CENT).

Species.	Locality.	Heart-wood.	Slab-wood.	Lum-ber.	Mean heart-wood and slab-wood.	Average lum-ber and heart-wood.	Other forms.
		<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
Beech.....	Indiana.....	1.95	1.79	2.04	1.87	1.99	{Bark..... 1.25 Sapwood... 1.97
Do.....	Pennsylvania.....	2.23	2.09		2.16		
Birch.....	Wisconsin.....	1.45	1.55	1.67	1.50	1.54	
Do.....	Pennsylvania.....	1.62	1.59		1.605		
Maple.....	Wisconsin.....	1.94	1.91	1.59	1.93	1.76	Bark..... 1.88
Do.....	Pennsylvania.....	1.94	1.78		1.86		
Red gum.....	Missouri.....	1.76	1.73		1.75		
Chestnut.....	New Jersey.....	.90	.87		.89		Limbs..... .96
Hickory.....	Indiana.....			2.08		2.08	
White oak.....	do.....	1.34	1.33	1.51	1.33	1.43	
Do.....	Arkansas.....	1.33	1.46		1.39		
Tupelo.....	Missouri.....	1.56	1.86		¹ 1.86		Limbs..... ¹ 1.64

YIELD OF ACETIC ACID (100 PER CENT).

Beech.....	Indiana.....	5.56	6.18	5.78	5.87	5.65	{Bark..... 2.98 Sapwood... 6.67
Do.....	Pennsylvania.....	5.77	6.21		5.99		
Birch.....	Wisconsin.....	6.71	6.88	6.62	6.80	6.68	
Do.....	Pennsylvania.....	6.19	6.10		6.15		
Maple.....	Wisconsin.....	5.42	5.11	5.58	5.24	5.49	Bark..... 3.15
Do.....	Pennsylvania.....	5.66	5.44		5.55		
Red gum.....	Missouri.....	5.70	5.23		5.46		
Chestnut.....	New Jersey.....	5.50	5.26		5.38		Limbs..... 6.42
Hickory.....	Indiana.....			5.05		5.05	
White oak.....	do.....	4.97	4.77	4.84	4.87	4.78	
Do.....	Arkansas.....	4.23	4.35		4.29		
Tupelo.....	Missouri.....	4.49	5.19		5.19		Limbs..... ¹ 5.64

¹ Heartwood not included in average, since only one distillation was made on this material.

The yields of alcohol and acetic acid vary a great deal among the different species, more so for alcohol than for acetic acid. A given species may rank high in its yield of alcohol but low in its yield of acid. Thus chestnut, which gives the lowest yield of alcohol, is among the highest in yield of acid; and hickory, which is among the highest in alcohol yield, is among the lowest in acid yield.

The average yield from the beech, birch, and maple wood from Wisconsin and Indiana is somewhat higher for acid and considerably lower for alcohol than for the same species in Pennsylvania. This difference, when figured to commercial products—namely, 82 per cent alcohol and 80 per cent acetate of lime—amounts to about 10 per cent of the alcohol and $1\frac{1}{2}$ per cent of the acetate of lime (see Table 4). The greatest differences are in the alcohol yield from beech and in the acid yield from birch. In the case of maple, the yields of both acid and alcohol are slightly higher from the Pennsylvania than from the Wisconsin wood. In contrast to these variances in absolute yield, the relative yield of the three species in either product does not change with the locality. The order of yield for alcohol is beech, maple, birch; for acid, birch, beech, maple. In the case of oak, the largest difference lies in the acid yield, the material from the more southern locality giving the lower yield. The yield of alcohol from wood cut in different States is very nearly the same, but if the runs on lumber are included the average is slightly higher for material from the northern localities.

VARIATION DUE TO FORM OF MATERIAL.

Although slabs with much bark are usually considered very poor material for distillation, the yields of alcohol and acetic acid from slabs having as high as 13 to 25 per cent bark by volume are in most cases only slightly lower, and in some cases even higher, than from heartwood. Distillation of beech bark showed that the higher yields of acid from beech slabs were not due to the bark, but to the very high yields of the sapwood (slabs without bark). These offset the low bark yields sufficiently to account for the fact that slabs with 13 per cent bark yielded more acid than the heartwood without bark. The same or higher yield of acid from the slabs of birch and tupelo and from the limbs of chestnut and tupelo is probably due to the same cause. The yields of alcohol from the sapwood of beech were practically the same as from the heartwood, and since the bark yields considerably less alcohol, the slabs with 13 per cent bark yielded less than the heartwood. Maple bark yielded very nearly as much alcohol as the heartwood, which accounts for the relatively high yields from the slabs.

CHARCOAL, TAR, AND GAS.

The yields of charcoal, tar, and gas are not included in Table 2, since they are influenced very much by the maximum temperatures of distillation and therefore are not comparable to the same extent as the alcohol and acetic-acid yields. Besides, these products are of indefinite composition, which further prevents comparison. There are some points of interest, however, in the relations between the yields of these products, and in Table 3 the average yields of alcohol, tar, and charcoal are shown, the species being arranged in order of the

yields of alcohol.¹ The total tar yields follow almost the same order as the alcohol yields. The yields of charcoal, on the other hand, tend to follow the reverse order, but with more exceptions. The lowest yield of charcoal and the highest yields of alcohol and of tar are obtained from one species—hickory; while the highest yield of charcoal and the lowest yields of alcohol and of tar are also obtained from one species—chestnut.

TABLE 3.—Average yields of alcohol, total tar, and charcoal from the heartwood of various species, in percentages of dry weight of material distilled.

Species.	Alcohol.	Total tar.	Charcoal.	Species.	Alcohol.	Total tar.	Charcoal.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Hickory.....	2.08	13.0	37.7	Tupelo.....	1.56	10.6	44.1
Beech.....	2.08	9.4	41.9	Birch.....	1.53	12.0	40.6
Maple.....	1.94	12.8	40.6	Oak.....	1.34	7.8	45.7
Red gum.....	1.76	11.7	38.6	Chestnut.....	.90	4.6	47.6

YIELDS PER CORD.

ALCOHOL AND ACETATE.

COMPARISON OF YIELDS.

Table 4 shows the same results as Table 2, but expressed in different units—the raw material in terms of gallons of 82 per cent wood alcohol and pounds of 80 per cent acetate of lime.

The yields from the various species on a cord basis are quite different from the yields on a unit weight basis; the heavier woods, such as hickory and oak, are advanced in relative position, and the lighter woods, such as chestnut and red gum, are reduced.

The average yield of alcohol from Indiana beech and Wisconsin birch and maple is 10.9 gallons per cord; the yield from these species from Pennsylvania is 11.51 gallons per cord. These figures represent the average yields obtained at commercial plants in these localities.² The average yield of acetate of lime from these two groups of woods, 319 pounds and 315 pounds per cord, respectively, is about 50 per cent higher than the average commercial yields. The yield from white oak from Arkansas of 9.2 gallons alcohol is very close to that being obtained in one commercial plant, and the acetate yield of 262 pounds per cord is, as in the case of the standard species, about 50 per cent higher than the commercial yield. The only ways in which the experimental distillations differed from commercial conditions were the low maximum temperatures and the short distance from the center of the charge to the heated surface of the retort. It is possible that these two conditions, resulting in a slow and well-controlled distillation, are sufficient to account for the higher yields.

¹ These averages do not include the yields from "lumber," since this material was usually very dry resulting in maximum temperatures much higher than the normal, giving yields of tar and charcoal not comparable with the rest of the runs.

² A corresponding difference between the Lake States and the Eastern States is also obtained commercially in the acetate yields, but this difference is not shown in the laboratory yields. It must be remembered, however, that these figures represent the average from equal proportions of the three standard species, while in actual practice one species may predominate.

In Table 5 are given the relative yields from the different forms and species, taking the average of the heartwood and lumber runs

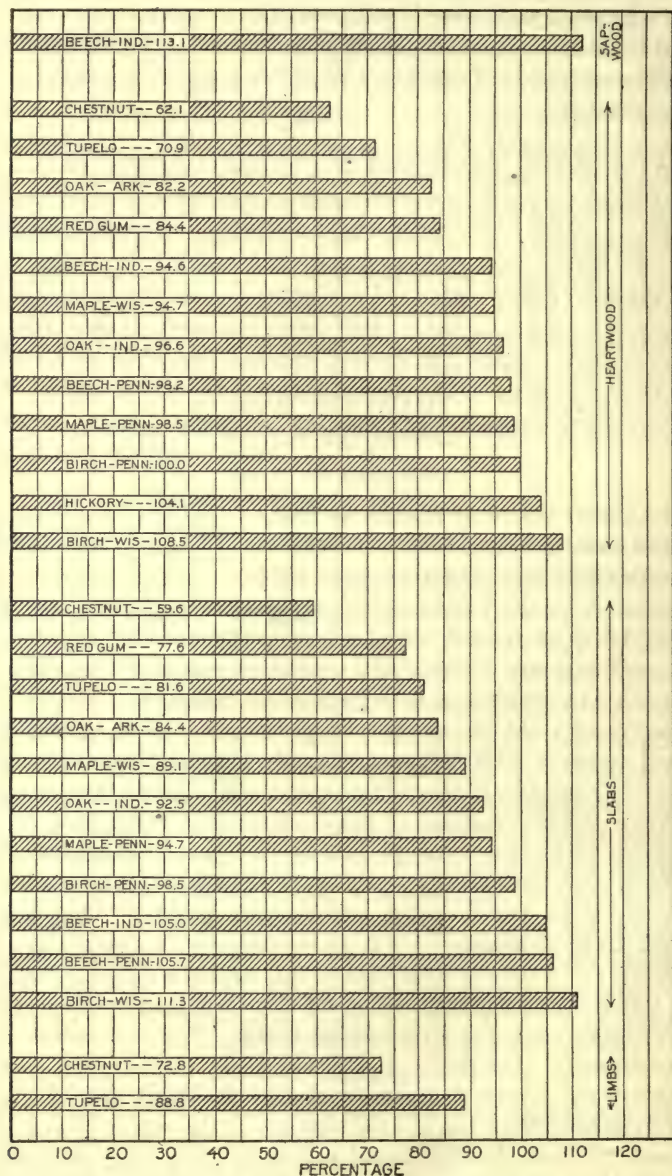


FIG. 2.—Relative yields of acetate of lime per cord. (Average yield from heartwood and lumber of beech, birch, and maple from Wisconsin=100 per cent.)

on beech, birch, and maple from Wisconsin as the standard (100 per cent). The same values are shown graphically in figures 2 and 3 for acetate and alcohol, respectively.

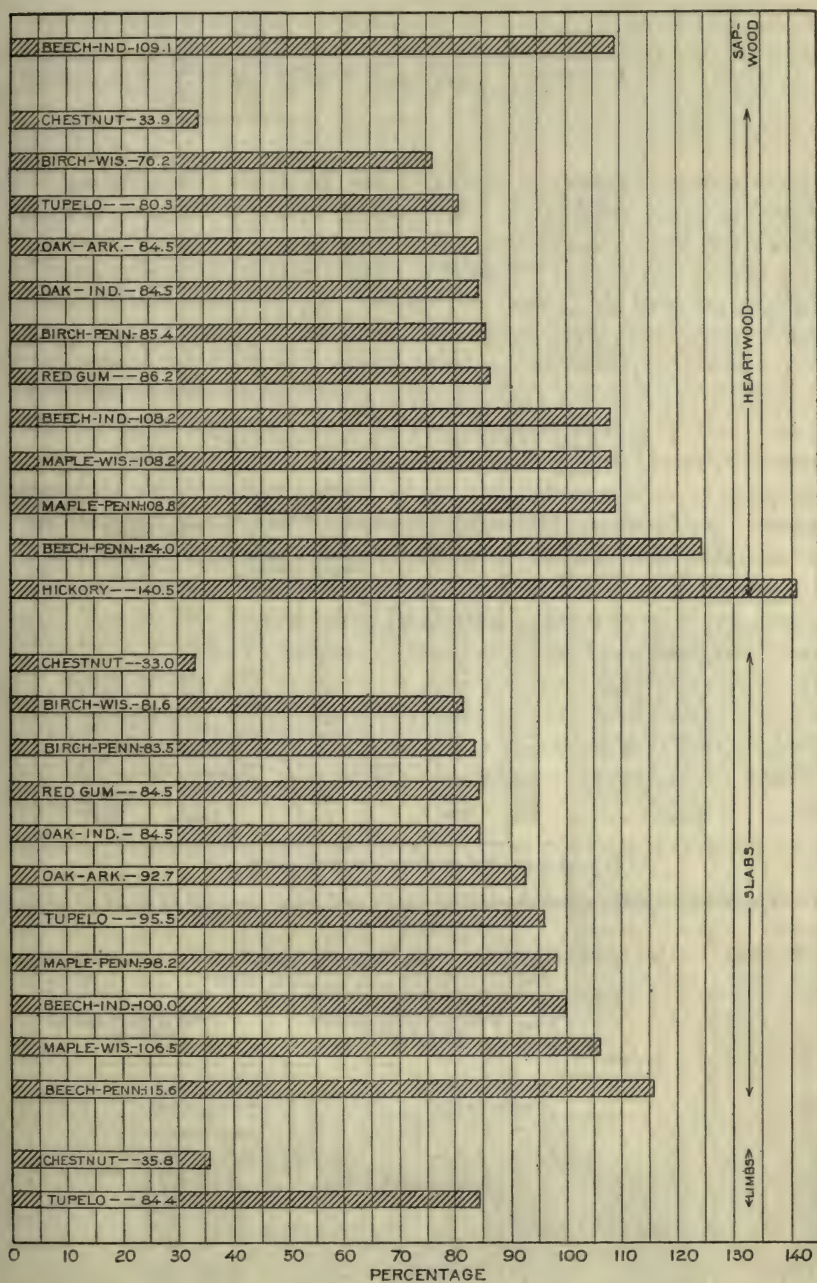


FIG. 3.—Relative yields of wood alcohol per cord of wood. (Average yield from heartwood and lumber of beech, birch, and maple from Wisconsin=100 per cent.)

TABLE 4.—*Yields of commercial alcohol and acetate from various species per cord of wood.*
YIELD OF WOOD ALCOHOL (82 PER CENT).

Species.	Locality.	Heart-wood.	Slab wood.	Lumber.	Mean heart-wood and slab wood.	Average lumber and heart-wood.	Other forms.
		<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>
Beech.....	Indiana.....	11.8	10.9	12.2	11.4	12.05	Sapwood... 11.9
Do.....	Pennsylvania.....	13.5	12.6		13.15		
Birch.....	Wisconsin.....	8.3	8.9	9.6	8.6	8.9	
Do.....	Pennsylvania.....	9.3	9.1		9.2		
Maple.....	Wisconsin.....	11.8	11.6	9.87	11.7	10.9	
Do.....	Pennsylvania.....	11.85	10.7		11.3		
Red gum.....	Missouri.....	9.4	9.2		9.3		
Chestnut.....	New Jersey.....	3.7	3.6		3.6		Limbs..... 3.9
Hickory.....	Indiana.....			15.3			
White oak.....	do.....	9.2	9.2	10.4	9.2	9.9	
Do.....	Arkansas.....	9.2	10.1		9.65		
Tupelo.....	Missouri.....	18.75	10.4		9.1		Limbs..... 9.2

¹ Single unchecked determination.

YIELD OF ACETATE OF LIME (80 PER CENT).

Species.	Locality.	Heart-wood.	Slab wood.	Lumber.	Mean heart-wood and slab wood.	Average lumber and heart-wood.	Other forms.	Weight per cord ¹ (15 per cent moisture).
		<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>
Beech.....	Indiana.....	301	335	313	318	307	Sapwood... 361	3,785
Do.....	Pennsylvania.....	313	337		325			3,785
Birch.....	Wisconsin.....	346	355	341	350	343		3,600
Do.....	Pennsylvania.....	319	314		316			3,600
Maple.....	Wisconsin.....	301	284	310	293	305		3,875
Do.....	Pennsylvania.....	314	302		308			3,875
Red gum.....	Missouri.....	269	247		258			3,300
Chestnut.....	New Jersey.....	198	190		194		Limbs..... 232	2,520
Hickory.....	Indiana.....			332		332		4,590
White oak.....	do.....	308	295	300	301	305		4,320
Do.....	Arkansas.....	262	269		265			4,320
Tupelo.....	Missouri.....	226	260		243		Limbs..... 283	3,510

¹ The weights per cord are derived as explained on p. 6.TABLE 5.—*Relative yields of commercial alcohol and acetate per cord of wood of various species.*¹[Average yields² from heartwood and lumber of beech, birch, and maple from Wisconsin equals 100 per cent.]

Species.	Locality.	Yield of acetate of lime (80 per cent).			Yield of wood alcohol (82 per cent).		
		Heart-wood.	Slab wood.	Other forms.	Heart-wood.	Slab wood.	Other forms.
		<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>	<i>Gals.</i>
Beech.....	Indiana.....	94.6	105.0	Sapwood... 113.1	108.2	100.0	Sapwood 109.1
Do.....	Pennsylvania.....	98.2	105.7		124.0	115.6	
Birch.....	Wisconsin.....	108.5	111.3		76.2	81.6	
Do.....	Pennsylvania.....	100.0	98.5		85.4	83.5	
Maple.....	Wisconsin.....	94.7	89.1		108.2	106.5	
Do.....	Pennsylvania.....	98.5	94.7		108.8	98.2	
Red gum.....	Missouri.....	84.4	77.6		86.2	84.5	
Chestnut.....	New Jersey.....	62.1	59.6	Limbs... 72.8	33.9	33.0	Limbs... 35.8
Hickory.....	Indiana.....	104.1			140.5		
White oak.....	do.....	96.6	92.5		84.5	84.5	
Do.....	Arkansas.....	82.2	84.4		84.5	92.7	
Tupelo.....	Missouri.....	70.9	81.6	Limbs... 88.8	80.3	95.5	Limbs... 84.4

¹ The weights per cord are derived as explained on p. 6.² Acetate of lime, 80 per cent, 319 pounds; alcohol, 82 per cent, 10.9 gallons.

COMPARISON OF VALUES.

The combined value of commercial alcohol and acetate of lime from the various forms of material per cord of wood of the various species is given in Table 6, which is computed from the yields given in Table 4. These values are according to the prevailing prices for 1911-13.¹

In judging the value of the different forms and species for distillation, the yields under commercial conditions must be considered, and therefore the value of the acetate per cord of wood of different species is computed on a basis of two-thirds of the yields shown. The value of the alcohol is based on the experimental yields, since these are at most only slightly higher than average commercial yields. Tar and gas are not included in the computation of the value of products, because they are relatively unimportant and are commonly used as fuel at the plant. Charcoal is one of the valuable commercial products, but is not included because there are no definite data from which the values might be computed. The charcoal produced by the experimental method has not been subjected to temperatures as high as in commercial practice, and therefore contains more volatile matter. Although the relation between the yields obtained from different species in the experiments might be the same as between those obtained by commercial methods, there is no information on the quality of the charcoal from different species. Also, charcoal is usually sold by volume, and since no data are available on the weight per bushel for that produced from different species, the yields can not be computed to commercial units.

TABLE 6.—*Values of commercial alcohol and acetate per cord of wood of various species.*¹

Species.	Locality.	Values of wood alcohol and acetate of lime during period 1911-13. ²						Weight of cord, ³ (15 per cent moisture).
		Heart-wood.	Slab-wood.	Lumber.	Mean heart-wood and slab-wood.	Other forms.	Average lumber and heart-wood.	
Beech.....	Indiana.....	\$8.08	\$8.41	\$8.38	\$8.26	Sapwood, \$9.10..	\$8.23	<i>Pounds.</i>
Do.....	Pennsylvania.....	8.72	8.89		8.81			3,785
Birch.....	Wisconsin.....	7.92	8.22	8.17	8.07		8.04	3,785
Do.....	Pennsylvania.....	7.73	7.59		7.66			3,600
Maple.....	Wisconsin.....	8.08	7.74	7.72	7.91		7.90	3,600
Do.....	Pennsylvania.....	8.31	7.80		8.06			3,875
Red gum.....	Missouri.....	6.92	6.50		6.71			3,875
Chestnut.....	New Jersey.....	4.28	4.11		4.19	Limbs, \$4.80..		3,300
Hickory.....	Indiana.....			9.51			9.51	2,520
Oak.....	do.....	7.52	7.30	7.70	7.41		7.61	4,590
Do.....	Arkansas.....	6.76	7.08		6.92			4,320
Tupelo.....	Missouri.....	6.04	7.03		6.59	Limbs, \$7.10..		4,320
								3,510

¹ The market price of crude alcohol is fairly stable, but acetate of lime fluctuates considerably from time to time. For this reason the relative value of the different species, from the standpoint of value of products, may vary from the calculations indicated.

² The weights per cord are derived as explained on p. —.

³ At 26 cents per gallon for alcohol and \$2.50 per hundredweight for acetate of lime. The acetate is computed from two-thirds the yields given in Table 4.

⁴ One determination only.

TABLE 7.—*Relative values of commercial alcohol and acetate per cord of wood of various species.*¹

[Average value of yields from heartwood and lumber of beech, birch, and maple from Wisconsin (\$8.06) equals 100 per cent.]

Species.	Locality.	Heart-wood.	Slabs.	Heart-wood and lumber.	Mean heart-wood and slabs.	Sapwood.	Limbs.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Beech.....	Indiana.....	100.2	104.2	102.1	102.5	113.0	
Do.....	Pennsylvania.....	108.1	110.2		109.2		
Birch.....	Wisconsin.....	98.4	102.0	97.7	100.1		
Do.....	Pennsylvania.....	96.0	95.5		95.8		
Maple.....	Wisconsin.....	100.2	96.0	95.8	98.1		
Do.....	Pennsylvania.....	103.0	97.0		100.0		
Red gum.....	Missouri.....	86.0	80.7		83.3		
Chestnut.....	New Jersey.....	53.2	51.0		52.1		60.7
Hickory.....	Indiana.....			118.0			
White oak.....	do.....	93.3	90.7	94.5	92.0		
Do.....	Arkansas.....	83.9	83.0		85.9		
Tupelo.....	Missouri.....	75.0	87.2		81.8		88.1

¹ The weights per cord are derived as explained on p. 6.

Assuming that the value of the charcoal and the cost of plant operation per cord of wood is the same for all species, the differences in the value of the alcohol and acetate produced by the various woods represent the differences in the value of these woods for distillation purposes.¹ The average value of the alcohol and acetate yields from Indiana beech and Wisconsin birch and maple heartwood is \$8.06 per cord. The values of these products from the heartwood of chestnut, red gum, tupelo (slabs), and southern and northern oak, are less than this amount by \$3.78, \$1.14, \$1.03, \$1.30, and \$0.54, respectively; from hickory (factory waste) the products are \$1.55 greater in value. The average price paid is only about \$3.50 per cord, and consequently the use of chestnut for this purpose is out of the question. Oak, tupelo, and red gum, under favorable conditions of supply and cost, might be used profitably, while hickory should command a very good price for this purpose. Since these deductions are based on the value of the chemical products they apply less strongly in case of plants making only a partial recovery of these products.

The value of alcohol and acetate from the different forms and species as given in Table 6 are compared in Table 7 by means of a standard value. This value is \$8.06, being the average value of beech (from Indiana), birch, and maple heartwood (from Wisconsin). This standard is taken as 100 per cent and the other values are ranked accordingly.

¹ The assumption in regard to the cost of operation will undoubtedly hold so far as the destructive distillation of the wood is concerned. However, the cost of the refining operations is approximately proportionate to the amount of crude pyroligneous acid produced; although this is variable, it bears some relation to the yield of refined products. The large amount of crude pyroligneous acid per cord of hickory would tend to increase the refining cost per cord of wood; likewise the low yields of crude pyroligneous acid from chestnut, tupelo, and red gum would tend to lower the cost of these woods. Therefore, the assumption made is not entirely correct, but the differences are not great enough to affect seriously the conclusions.

The value of the two products (alcohol and acetate) from hickory is 18 per cent greater than the standard chosen for the comparison. Of the other species, oak from Indiana is the only one which falls above 90 per cent; with all the others, except chestnut, the average yield from heartwood and slab wood is above 80 per cent.

Of equal interest to these relative values based upon species are the relative values of the different forms of wood from the same species. These relations are shown in Table 8, in which the value of heartwood in each case is taken as 100 per cent. A number of species show a higher value for slabs than for heartwood. The slabs of Indiana beech, Wisconsin birch, Pennsylvania beech, and Arkansas oak are from 2 to 5 per cent higher than the heartwood, while the limbs of the two species tested, chestnut and tupelo, are about 15 per cent higher than heartwood. These results are based upon equal weights of the several forms of material compared.

TABLE 8.—*Relative values of commercial alcohol and acetate from equal weights of various forms of material.*

[Heartwood=100 per cent.]

Species.	Locality.	Heartwood.	Slab wood.	Sapwood.	Limbs.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Beech.....	Indiana.....	100	104.0	112.5	
Do.....	Pennsylvania.....	100	102.0		
Birch.....	Wisconsin.....	100	103.8		
Do.....	Pennsylvania.....	100	98.2		
Maple.....	Wisconsin.....	100	95.8		
Do.....	Pennsylvania.....	100	93.9		
Red gum.....	Missouri.....	100	94.0		
Chestnut.....	New Jersey.....	100	96.0		114.2
Hickory.....	Indiana.....	100			
White oak.....	do.....	100	97.2		
Do.....	Arkansas.....	100	104.8		
Tupelo.....	Missouri.....	100	116.5		117.5

PYROLIGNEOUS ACID, TAR, AND CHARCOAL.

The average yields of pyroligneous acid, tar, and charcoal from the various forms of material, expressed in pounds per cord for each species, are given in Table 9. Although the yields of these products, especially of the last two, are directly affected by the maximum temperatures of distillation, and are therefore not as accurate as the alcohol and acetate yields, some conclusions of interest can be drawn from them. The yields of pyroligneous acid are of interest mainly in connection with the cost of refining the products from a cord of wood. (See footnote on p. 14.)

The average commercial yield of charcoal from a cord of beech, birch, and maple is about 50 bushels or (at 20 pounds, the usual weight per bushel) 1,000 pounds; the average yield from the heartwood of the three species by the experimental method is 1,378 pounds per cord. This large difference is probably due chiefly to the low maximum temperatures of distillation, resulting in a charcoal with a high amount of volatile matter. Charcoal of this composition would probably be satisfactory as a fuel for domestic use, but where

high carbon content or high crushing strength is required it might not be suitable.

The yields of tar are also somewhat higher than those usually obtained in practice, and this can not be explained entirely by the low maximum temperature of distillation, since further distillation of the charcoal at higher temperatures gave increased production of tar. (See p. 4.) It is probable, however, that under the experimental conditions of distillation there was less tar decomposed into gas and coke than under commercial conditions where part of the tar would be subjected to considerably higher temperatures after formation.

TABLE 9.—Average yields per cord of pyroligneous acid, charcoal, and tar from various species.¹

PYROLIGNEOUS ACID (MINUS MOISTURE).

Species.	Locality.	Heart-wood.	Slab wood.	Lumber.	Mean heart-wood and slab wood.	Average lumber and heart-wood.	Other forms.
		<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>
Beech.....	Indiana.....	1,062	1,165	1,119	1,113	1,085	Sapwood.. 1,149
Do.....	Pennsylvania.....	1,180	1,158		1,169		
Birch.....	Wisconsin.....	1,152	1,159	1,220	1,156	1,167	
Do.....	Pennsylvania.....	1,249	1,135		1,192		
Maple.....	Wisconsin.....	1,120	1,061	1,207	1,145	1,157	
Do.....	Pennsylvania.....	1,125	1,164		1,145		
Red gum.....	Missouri.....	1,098	917		1,005		
Chestnut.....	New Jersey.....	790	644		716		Limbs.... 714
Hickory.....	Indiana.....			1,495		1,495	
White oak.....	do.....	1,230	1,170	1,275	1,200	1,156	
Do.....	Arkansas.....	1,155	1,120		1,135		
Tupelo.....	Missouri.....	1,081	1,065		1,073		Limbs.... 1,049

CHARCOAL.

Beech.....	Indiana.....	1,417	1,297	1,385	1,357	1,403	Sapwood.. 1,470
Do.....	Pennsylvania.....	1,335	1,383		1,359		
Birch.....	Wisconsin.....	1,315	1,284	1,286	1,300	1,303	
Do.....	Pennsylvania.....	1,228	1,300		1,265		
Maple.....	Wisconsin.....	1,341	1,515	1,348	1,344	1,430	
Do.....	Pennsylvania.....	1,352	1,268		1,310		
Red gum.....	Missouri.....	1,058	1,360		1,210		
Chestnut.....	New Jersey.....	1,041	1,160		1,102		Limbs.... 1,061
Hickory.....	Indiana.....			1,500		1,500	
White oak.....	do.....	1,858	1,892	1,625	1,875	1,715	
Do.....	Arkansas.....	1,580	1,734		1,654		
Tupelo.....	Missouri.....	1,400	1,405		1,402		Limbs.... 1,320

TOTAL TAR.

Beech.....	Indiana.....	319	349	342	332	329	Sapwood.. 250
Do.....	Pennsylvania.....	299	359		329		
Birch.....	Wisconsin.....	325	285	338	307	330	
Do.....	Pennsylvania.....	426	347		385		
Maple.....	Wisconsin.....	418	310	500	450	360	
Do.....	Pennsylvania.....	422	416		418		
Red gum.....	Missouri.....	336	215		276		
Chestnut.....	New Jersey.....	102	80		91		Limbs.... 173
Hickory.....	Indiana.....			519		519	
White oak.....	do.....	237	173	331	203	285	
Do.....	Arkansas.....	349	327		338		
Tupelo.....	Missouri.....	348	370		364		Limbs.... 447

¹ The weights per cord were derived as explained on p. 6.

Walter Muirford

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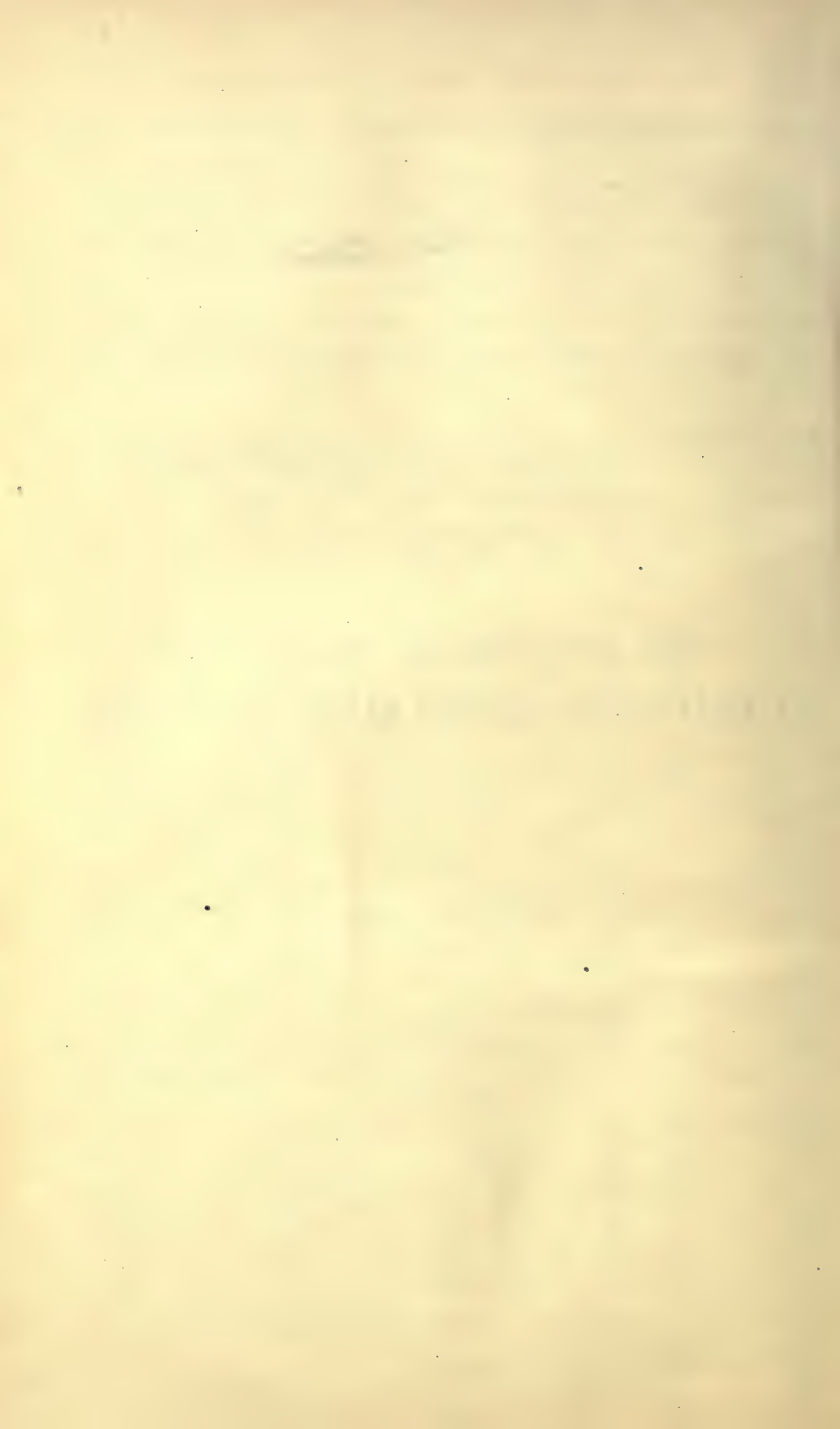
GIFFORD PINCHOT, Forester.

THE ANALYSIS OF TURPENTINE
BY FRACTIONAL DISTILLATION WITH STEAM.

By

WILLIAM C. GEER,

EXPERT.



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THE ANALYSIS OF TURPENTINE BY FRACTIONAL DISTILLATION WITH STEAM.

FORMER METHODS OF TURPENTINE ANALYSIS.

In the course of the studies by the Forest Service concerning the utilization of waste yellow pine wood by distillation processes,^a it was found that the "wood turpentines" produced by these methods were extremely variable in composition and properties, and that an accurate method for comparing and grading them was very necessary for the further progress of the work. Most of the methods of turpentine analysis in use were devised simply to detect adulterations in gum turpentine, and there was no satisfactory method applicable to all classes of turpentines which would give an accurate idea of their relative compositions and characteristics. Fractional distillation with subsequent examination of the individual fractions appeared the best for this purpose, but some modifications seemed advisable. This general method is ordinarily carried on as follows: The liquid to be distilled is heated in a flask, the vapors are condensed, and the distillate is collected in several portions or fractions; the temperatures between which the various fractions distill are recorded, and sometimes one or more physical properties of the fractions are determined. In this manner a general idea of the composition of a turpentine can be obtained, but, as will be shown later, the completeness of the separation, the ease of interpretation of the results, and the reliability of the data can be increased by extending and modifying the ordinary method.

MODIFICATIONS INTRODUCED.

The four following important changes were made:

- (1) A new still head was introduced.
- (2) Steam distillation was used instead of the ordinary distillation with direct heat.
- (3) The graphical method of expressing results was employed.
- (4) More physical properties were determined.

These changes will be discussed in order.

- (1) If a mixture of two liquids with different boiling points be distilled, each of the first fractions will contain a larger proportion of the

^a Forest Service Circular 114, "Wood Distillation."²²

lower boiling liquid, and each of the last a larger proportion of the higher boiling liquid; all the fractions will contain both liquids in varying proportions, and it will be impossible to separate the two liquids completely by one such distillation. By repeated distillations of the several fractions, however, and by combination of those fractions which distill between the same temperatures, a fairly complete separation can eventually be made. This is usually a long and tedious operation and not readily applicable to an analytical method.

By taking advantage of the well-known principle of dephlegmation these difficulties are largely removed and in many cases one operation will suffice to separate two liquids in practically pure condition. This principle is applied by placing between the distilling flask and the condenser a new form of column still head or dephlegmator (fig. 4). This is of such construction that the vapors are partly condensed before they reach the condenser, and the uprising vapors are therefore washed by the reflowing liquid. When a mixture of light and heavy^a oils, such as might occur in turpentines, is distilled through this apparatus, the tendency is for the vapors of the light oil to pass freely out into the condenser, while the vapors of the heavy oil are condensed and return to the still, to be carried over only after all the light oil has been distilled. The results are the same as though repeated distillations were made.

(2) When distilling high boiling oils over a direct flame, it is difficult to regulate the temperature, and there is always danger of superheating. It is true that this superheating may be slight when the distillation is carefully carried on, but if the operation is hastened the danger may be greatly increased. In the case of turpentine and other liquids which contain unstable substances, like terpenes, superheating may cause decomposition or alteration of the oil, and for this reason a study of the properties of the distillate may not clearly show the composition of the original oil.

Not only does superheating tend to cause decomposition of the turpentine, but it introduces errors in temperature readings. Further, the rate of distillation may not be constant. These several sources of error involved in an analysis by ordinary direct heat distillation can be largely eliminated by distillation with steam, since the process can be carried on at a much lower temperature and with a more certain regulation.

The fundamental principles of distillation with steam may be outlined as follows: If a pure liquid, such as benzine, which does not dissolve in water be placed in a flask and water added, the two will upon

^a "Light" and "heavy" are terms used synonymously with low boiling and high boiling. The terms arise from light and heavy weight—that is, low and high specific gravity which usually accompany low and high boiling points respectively.

the application of heat distill together in a fixed proportion, and the boiling point will remain unchanged as long as the two constituents of the mixture are present. This boiling will take place at a temperature below the boiling point of either the oil or the water. This lowering of boiling point is characteristic of a mixture of two mutually insoluble liquids, and because of this fact an oil with a high boiling point may be distilled with water at a temperature below the boiling point of the latter. The same conditions exist and the same results follow when saturated steam is blown into the oil. In practice the steam distillation is carried on either by mixing the water with the oil in the still and heating the still externally from beneath, or by blowing steam into the oil from a boiler; the latter process is easier to regulate.

If, however, nearly equal quantities of two oils which are freely miscible with one another and have different boiling points be mixed and distilled with steam, the proportion of oil and water constantly changes and the distillation temperature of such a mixture does not remain constant, but will vary between the approximate temperature at which the separate oils distill with steam.

The separation of light and heavy oils by steam distillation offers the same difficulties as were mentioned in the discussion of direct heat fractionation.

To remove these difficulties the principles of dephlegmation can be applied to steam distillation in the same manner as to ordinary distillation, but the still head must be of special construction.

(3) In scientific work of this character it is often very difficult, even with the data in hand, to interpret the results quickly and accurately. This is due in part to the fact that the eye can not readily grasp the meaning of a mass of figures. They are too detailed, and too much calculation is necessary in order to make comparisons. Something less complicated would greatly aid matters; a picture, as it were, which represents the exact conditions.

If in the case of an ordinary distillation without a well made still head the maximum boiling points of the various fractions be plotted on the horizontal axis, and the corresponding percentages by weight of distillate on the vertical axis, a curve which approximates figure 1 will

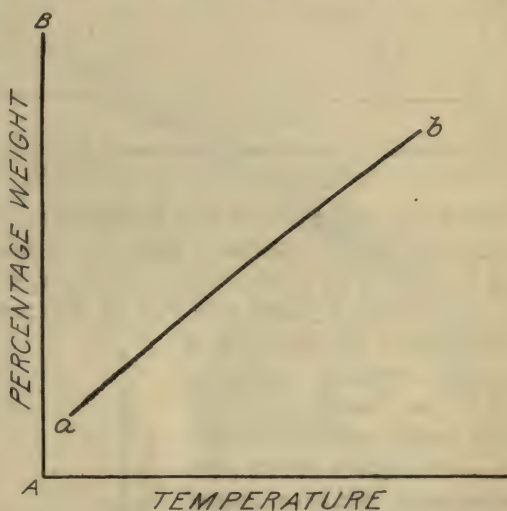


FIG. 1.—Percentage weight-steam temperature curve for a mixture of two oils distilled in the ordinary manner.

be obtained, which shows graphically what has happened during distillation. This curve shows that with the increase in the total percentage of oil distilled, the

boiling temperature gradually rose, indicating that the proportion of the higher boiling oil increased in the later fractions. It also shows that the two oils are mixed in the various fractions in varying proportions.

If, however, the same mixture is distilled through a dephlegmator, the percentage weight-boiling temperature curve will take a form similar to that shown in figure 2. In this figure the curve shows that

during the first part of the distillation from *a* to *b* the temperature remained nearly constant, from *b* to *c* the temperature rose rapidly, while from *c* to *d* the temperature was again practically constant and remained so until the end of the process. It is evident, there-

fore, that a curve which is nearly vertical indicates that the distillate during that portion of the distillation consists of a nearly pure substance, while a curve which rises slowly toward the right or is nearly horizontal indicates that the distillate is composed of two or more substances. A comparison of figure 1 with figure 2 shows clearly the more complete fractionation due to dephlegmation, and also shows that when dephlegmation is used the shape of the curve gives a more accurate picture of the composition of the mixture distilled.

Figure 3 is the type of curve obtained when the oil distilled consists chiefly of one substance with a relatively small admixture of higher boiling substances.

[Cir. 152]

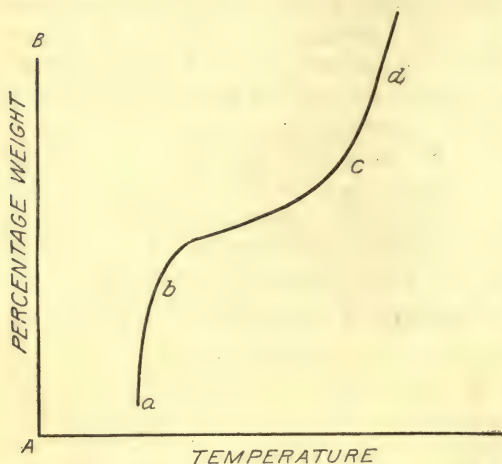


FIG. 2.—Percentage weight-steam temperature curve for a mixture of two oils distilled through a dephlegmator.

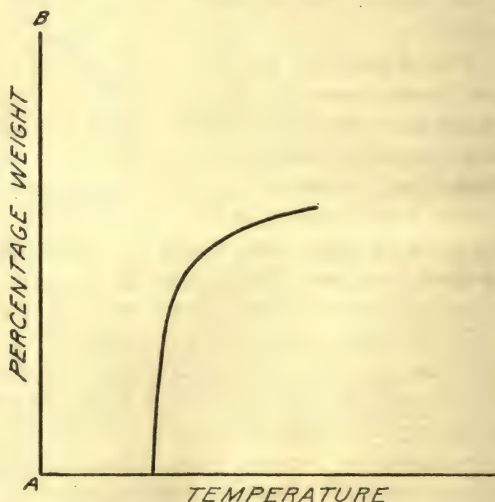


FIG. 3.—Percentage weight-steam temperature curve for a nearly pure oil mixed with small quantities of higher boiling substances distilled through a dephlegmator.

(4) Any physical property, such as specific gravity or index of refraction, may be used in the place of boiling temperature in these curves; and the more physical properties determined, the greater will be the reliability of the final interpretation. In the analyses made by the Forest Service, the color, odor, and maximum boiling temperature of all fractions were noticed, and specific gravity and index of refraction were determined.

THE NEW METHOD OF ANALYSIS.

The complete method as finally modified consists, then, in distilling the sample with steam through a dephlegmator and collecting the dis-

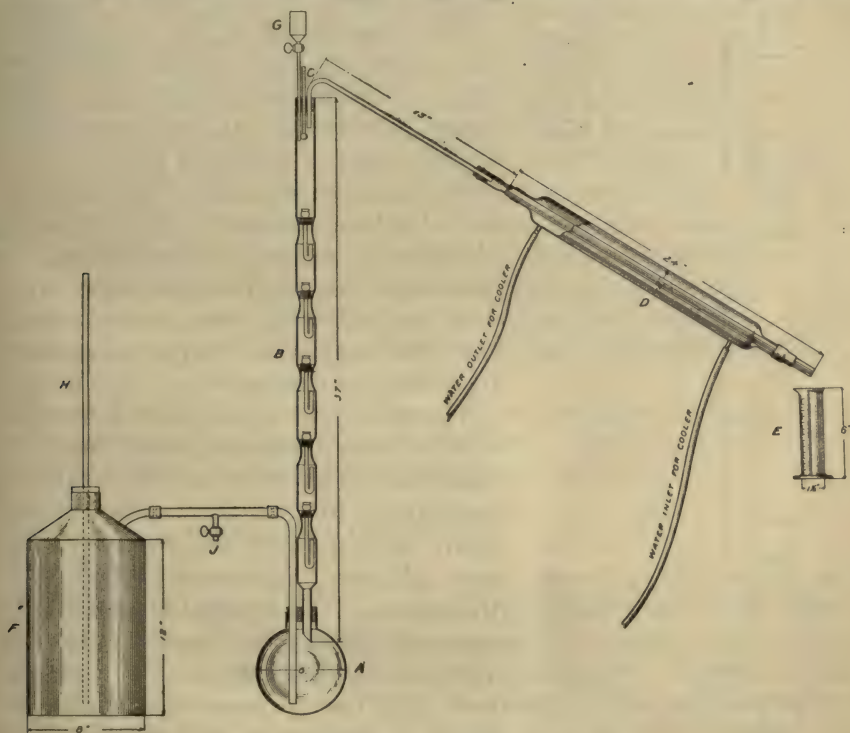
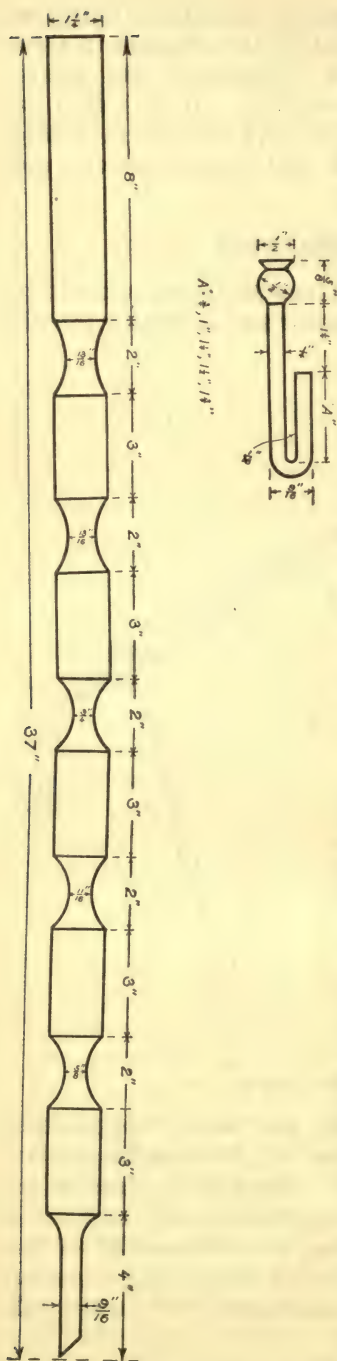


FIG. 4.—Apparatus used in turpentine analysis.

tillate in several fractions. The color, odor, and boiling temperature of each of these fractions are noted, and the weight, specific gravity, and index of refraction of each determined. From these data, tables are made and curves are plotted. The interpretation and comparison of the curves furnish information concerning the composition of the samples as shown by the physical properties of the leading constituents, and also render the similarity or dissimilarity of various oils quickly evident.

APPARATUS.

The apparatus, shown in figure 4, is supported by the ordinary laboratory stands and clamps, which are omitted in the drawing. A



lars instead of through the tube itself. The bulbs at the upper ends of the U tubes prevent them from falling, and the bends are made in order to hold a small amount of liquid, and thus act as a trap to prevent the escape of vapors through the tubes instead of through the gauze. Each trap was one-fourth inch shorter than the one below it, since it was found advisable to have successively heavier water seals from top to bottom. Each constriction was made smaller than the one above it, with the exception of the two at the top, in order to allow the collars to be easily placed in position when the dephlegmator is being constructed. A strip of 40-mesh brass gauze^a three-eighths of an inch wide was carefully wrapped about each tube until the size of the collar was sufficient to fit its particular constriction snugly. The U was then dropped carefully into the large tube and gently forced into position with a glass rod. A snug but not tight fit is desired to prevent the tubes from blowing out of position while the apparatus is in operation and at the same time to permit of easy withdrawal for cleaning. To facilitate removal, a loop of copper wire is fastened to the top of the tube into which a hook can be inserted. In this way each section can be readily removed. It is a matter of some skill to set up the dephlegmator, but when the apparatus is once in position it is ready for a long series of determinations. Cleaning, in the case of turpentine distillations, can be done by ordinary solvents, such as alcohol and ether, without taking the apparatus apart.

Before use each time it is necessary to see that the bent tubes inside the dephlegmator are filled with water. This is done by pouring water into the top and allowing it slowly to run out.

OPERATION.

The operation of the apparatus is simple. The boiler is about half filled with water, and the separatory funnel *G* is entirely filled. The flask *A*, previously dried and weighed, is placed on a balance capable of weighing accurately to 10 mg., and exactly 500 grams of the sample of turpentine is poured in. The flask is then replaced in the position shown in the diagram. A sample of the original oil should be reserved for comparison with the several fractions of the distillate. The burner under the boiler is then lighted, and when the steam is freely escaping through the valve *J*, the valve is closed to allow the vapor to pass into *A* and the burner turned down to a point determined by experience. It is necessary to have the distillation carried on at a slow and uniform rate. Two drops of distillate per second is the rate that has been found to give the best results. In order to keep this rate constant all heat conditions must be carefully regulated. The flame under *A* should be small and low to prevent excessive con-

^a Platinum could, of course, be used to distill liquids that would attack brass.

[Cir. 152]

densation, and the burners should be surrounded by asbestos shields to keep off drafts of air. The water in *G* also is allowed to drip slowly, at the rate of about one drop per second, to insure plenty of liquid on the brass collars.

At the beginning of the distillation it is necessary to watch the still rather carefully and occasionally to relieve the steam pressure through the valve *J*, until the dephlegmator is evenly heated and the rate of reflow uniform in each section.

It is customary, in ordinary fractional distillation, to change the receiver at regular temperature intervals, which have been previously determined. In this method, however, the receiver is not changed according to prescribed temperature intervals, but according to the nature of the distillation. If the oil contains a large proportion of one substance, the temperature will remain nearly constant while that portion of the oil is being distilled. During this part of the distillation but few fractions need be taken. When, however, the oil is complex and the thermometer is rising rather rapidly, it is better to take more frequent and smaller fractions. Each time the cylinders are changed the temperature is recorded. Usually from ten to fifteen fractions are collected during one distillation. The cylinder with its contents is weighed and the weight of the dry cylinder subtracted. This gives the weight of the total distillate, which consists of both oil and water. Since by this method no greater accuracy than to 10 mg. is essential, the volume of the water in cubic centimeters as read on the cylinder can be assumed to be numerically equal to the weight of the water in grams. Subtracting this from the weight of the total distillate gives the weight of the turpentine in the fraction. By such a procedure results are obtained from which a table can be made giving for each fraction the steam temperature and the weight of oil it contains.

The odor and color of each fraction are observed and recorded at once. The specific gravity is determined, according to accepted methods, with a 10 c. c. pycnometer. The determinations are made at ordinary temperatures and corrected to give the value at 15° C. If the temperature at which the determinations are made is above 15°, then the correction factor 0.00083 is added for every degree of difference. If the temperature at which the determinations are made is below 15°, then 0.00083 is subtracted for every degree of difference. This correction factor was obtained experimentally by using a very pure wood turpentine. The index of refraction is read by means of an Abbe refractometer compensated to the D line of the spectrum, and the values corrected to 15° C. by means of factors. The factor 0.00049 is applied to all observed values below 1.4750, and is added for each additional degree of temperature above 15° C. and subtracted for each below. For oils having an index of refraction above 1.4750 the correction factor 0.00044 is similarly used. These values were

determined on two pure fractions of wood turpentine with indices of refraction above and below 1.4750, respectively.

There is now for each fraction the steam temperature, the weight, color, odor, specific gravity, and index of refraction.

RESULTS.

The results of the analyses of four different turpentines are given in the accompanying tables and curves. In the tables the data, at the top show the properties of the original sample; the first column gives the serial number of the fraction; the second, the steam temperature after correction for barometric fluctuations; the third, the weight of the turpentine in each fraction. The fourth column is made by adding the weight of each turpentine fraction to the weight of all the preceding turpentine fractions, which gives the total weight of oil distilled up to each point. Thus in Table 1 up to and including fraction 3, the total weight of turpentine distilled was 267.66 grams. After all the fractions had been collected, the total weight distilled was 493.85 grams, and the undistilled residue amounted to the difference between the original weight taken, 500 grams, and the total weight distilled, 493.85 grams, or 6.15 grams. For purposes of reference and calculation it is more convenient to deal with percentage weight than with total weight, and hence the fifth column is prepared from the fourth by finding the percentages which each total weight is of the original sample. The sixth and seventh columns give the corrected values for the specific gravity and index of refraction of each fraction. From these data three curves are drawn, showing the variation of steam temperature, specific gravity, and index of refraction, respectively, with the percentage weight of the distillate.

TURPENTINE NO. 19—A GUM TURPENTINE.

Table 1 and figures 6, 7, and 8 give the data obtained from the analysis of a gum turpentine according to the methods which have been described.

TABLE 1.—*Turpentine No. 19—A gum turpentine.*

[Specific gravity, 0.8693 at 15°; index of refraction, 1.4739 at 15°; weight of sample, 500 grams.]

No. of fraction.	Steam temperature corrected.	Weight of turpentine in fraction.	Total weight.	Percentage weight.	Specific gravity at 15° C.	Index of refraction at 15° C.
	° C.	Grams.	Grams.	Per cent.	Sp. g.	N D.
1.....	94.9	52.98	52.98	10.6	0.8668	1.4718
2.....	94.9	53.03	106.01	21.2	.8668	1.4717
3.....	95.0	53.02	159.03	31.8	.8670	1.4722
4.....	95.0	53.05	212.08	42.4	.8677	1.4724
5.....	95.0	55.58	267.66	53.5	.8674	1.4727
6.....	95.0	52.45	310.11	62.0	.8674	1.4730
7.....	95.2	52.35	362.46	72.5	.8675	1.4733
8.....	95.2	51.92	414.38	82.9	.8682	1.4740
9.....	95.7	49.57	463.95	92.8	.8684	1.4757
10.....	98.7	29.90	493.85	98.8	.8866	1.4849

The ten points indicated on the curve in figure 6 show the specific gravity of the ten fractions and the corresponding percentage weight

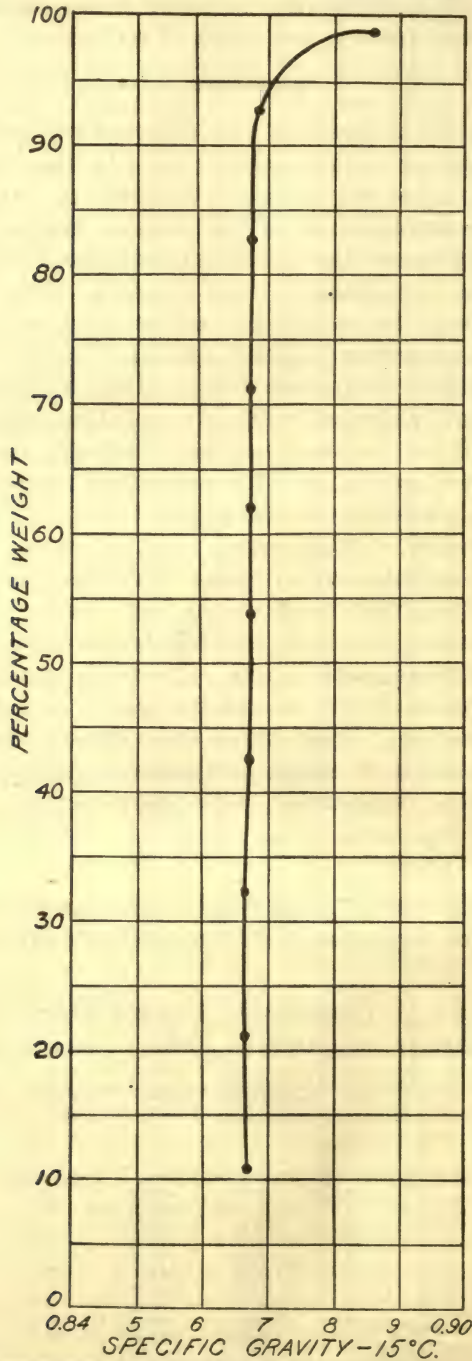


FIG. 6.—Percentage weight-specific gravity curve for a gum turpentine.

of each fraction. The specific gravity of the first 92½ per cent distilled is seen to be very uniform, as it varied only from 0.8660 to

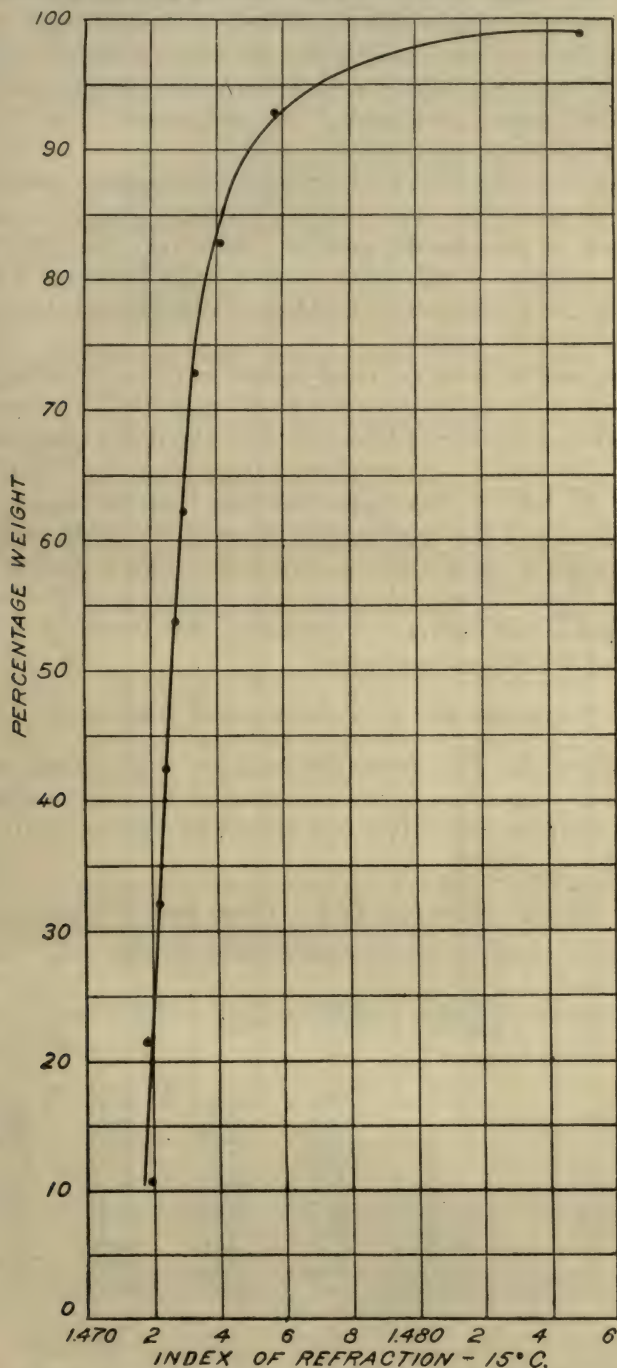


FIG. 7.—Percentage weight-index of refraction curve for a gum turpentine.

0.8685, which is .0025, or 0.29 per cent. The $6\frac{1}{2}$ per cent between $92\frac{1}{2}$ and 99 per cent varied widely in specific gravity, while the last 1 per cent remained undistilled. Whether this $92\frac{1}{2}$ per cent was composed entirely of one substance, the curve does not decide, for it is possible that two substances of the same specific gravity were present. The term "uniformity of composition" in this paper refers only to physical properties.

On reference to the index of refraction-percentage weight curve, figure 7, it is seen that this physical property varied in about the same manner as the specific gravity. Over the first $92\frac{1}{2}$ per cent distilled, the index of refraction varied only between 1.4718 and 1.4757, 0.0039, or 0.27 per cent, and beyond $92\frac{1}{2}$ per cent the refractive index increased very rapidly.

The steam temperature curve as shown in figure 8 indicates about the same proportion of uniform composition, since the abrupt change in the direction of the curve takes place at about $92\frac{1}{2}$ per cent. Over this range the steam temperature varies from 94.9° to 95.7° C. About $92\frac{1}{2}$ per cent of this turpentine was therefore nearly uniform in composition, and the remainder consisted of a mixture of heavier oils. The original sample was colorless and had a sweet, pine-like odor. Each fraction was colorless and had a pine-like odor, and only the residue was yellow. Practically, Turpentine No. 19 would be considered a high-grade product.

TURPENTINE NO. 14—A CRUDE STEAM TURPENTINE.

Table 2 gives the data from the analysis of an unrefined, steam distilled, wood turpentine. The specific gravity, index of refraction, and steam temperature curves are shown in figures 9, 10, and 11, respectively.

TABLE 2.—*Turpentine No. 14—A crude steam turpentine.*

[Specific gravity, 0.8834 at 15° ; index of refraction, 1.4749 at 15° ; weight taken, 500 grams.]

No. of fraction.	Steam temperature corrected.	Weight of fraction.	Total weight.	Percentage weight.	Specific gravity at 15° C.	Index of refraction at 15° C.
	$^{\circ}$ C.	Grams.	Grams.	Per cent.	Sp. g.	N D. ⁺
1.....	95.1	32.92	32.92	6.6	0.8665	1.4701
2.....	95.1	51.86	84.78	18.0	.8664	1.4704
3.....	95.1	44.39	129.17	25.8	.8668	1.4704
4.....	95.2	42.64	171.81	34.3	.8666	1.4709
5.....	95.3	46.07	217.88	43.6	.8666	1.4709
6.....	95.5	45.34	263.22	52.6	.8668	1.4710
7.....	95.8	43.82	307.04	61.4	.8675	1.4718
8.....	96.1	29.02	336.06	67.2	.8685	1.4729
9.....	96.9	26.71	362.77	72.5	.8707	1.4744
10.....	97.7	22.12	384.89	77.0	.8766	1.4771
11.....	98.3	14.69	399.58	79.9	.8984	1.4809
12.....	98.7	12.41	411.99	82.4		1.4852

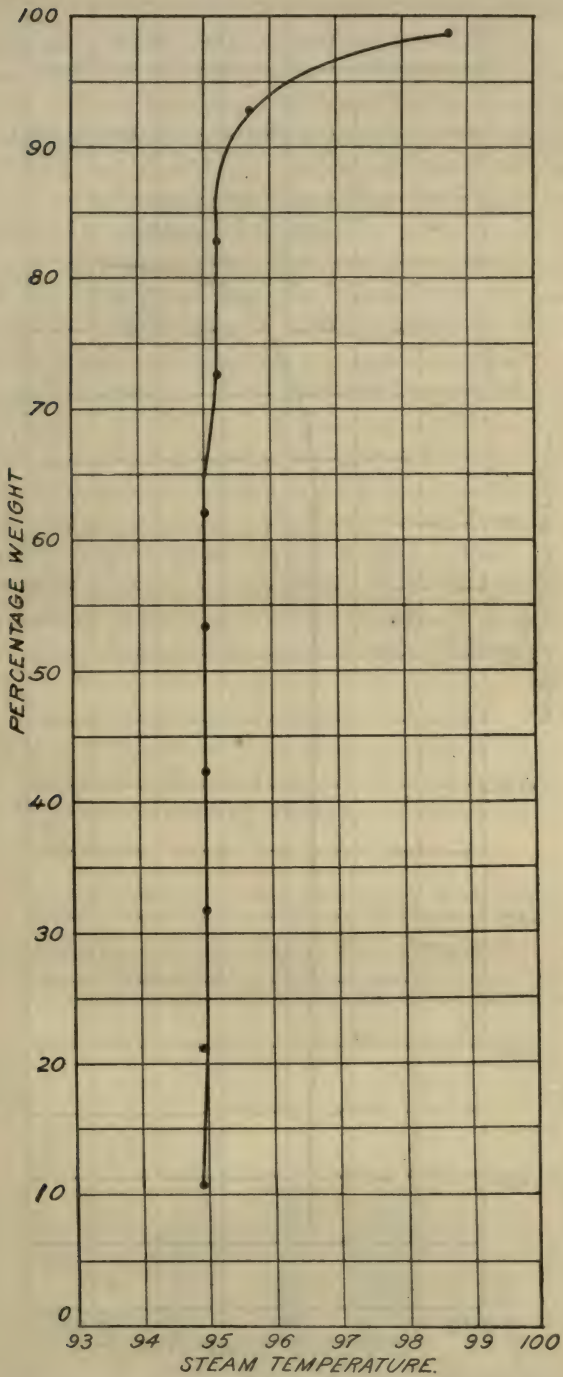


FIG. 8.—Percentage weight-steam temperature curve for a gum turpentine.
[Cir. 152]

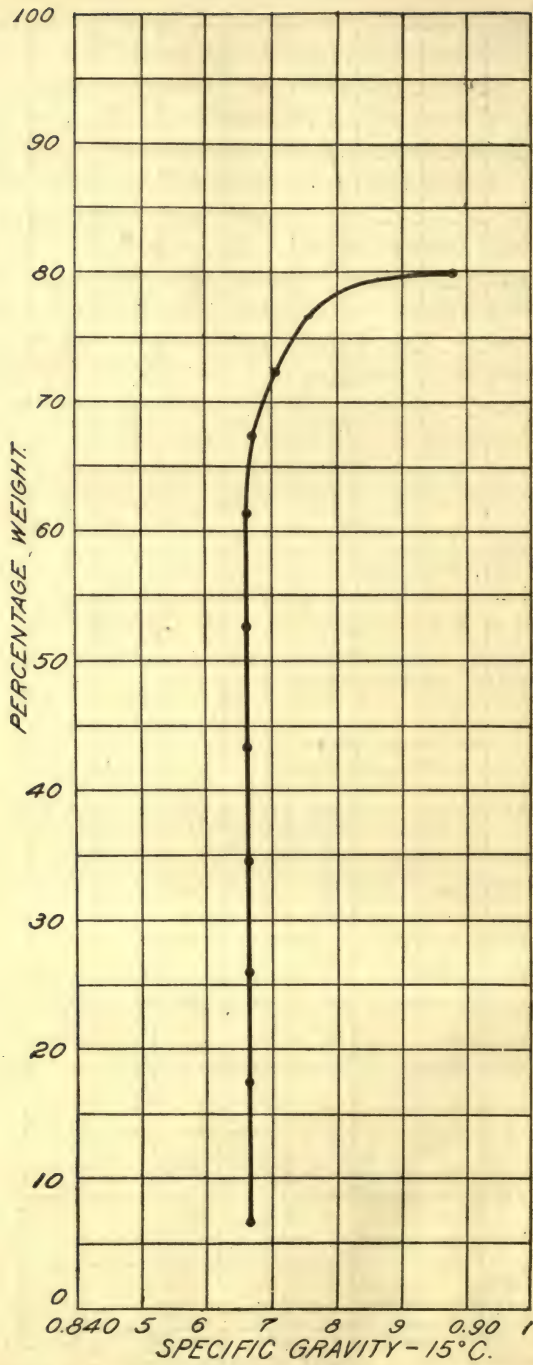


FIG. 9.—Percentage weight-specific gravity curve for a crude steam-distillation turpentine
[Cir. 152]

The specific gravity curve of this turpentine, figure 9, is similar in general respects to that of the gum turpentine of Table 1. It is seen, however, that about 70 per cent of the oil has a specific gravity within a variation of 0.0025 or between 0.8665 and 0.8690, a difference of 0.29 per cent. Above this the curve turns sharply to the right and ends at 80 per cent; the balance of 20 per cent was a heavy residue which was left in the flask. It seems, therefore, from the figure that this turpentine contained 70 per cent of a pure oil.

The index of refraction curve, figure 10, confirms this conclusion. Here we find that up to 70 per cent the index of refractions varied from 1.4701 to 1.4735, a difference of 0.0034, which is 0.23 per cent, or slightly less than the variation in the specific gravity over 92½ per cent of the gum turpentine. The distillate beyond 70 per cent was of a complex character, as is shown by the shape of the curve.

The form of the steam temperature curve, figure 11, is also similar to that showing the index of refraction. It rises nearly straight at first, but turns abruptly toward the right at about 70 per cent. After combining the interpretations of the several curves, it can be concluded with safety that the oil consisted of 70 per cent of a substance of uniform physical properties mixed with about 30 per cent of complex heavier oils. The original sample was a yellowish red in color, the fractions to 70 per cent were colorless and of a pine-like but not sweet odor; from 70 to 80 per cent they were light yellow, and the residue was red and rather strong smelling. These facts lead one to believe that Turpentine No. 14 is rather poor in quality. However, by suitable methods of refining 70 per cent of a high-grade product could be made from it.

TURPENTINE NO. 27—A REFINED STEAM TURPENTINE.

Table 3 gives the data for a refined steam turpentine.

TABLE 3.—*Turpentine No. 27—A refined steam turpentine.*

[Specific gravity, 0.8698 at 15°; index of refraction, 1.4709 at 15°; weight taken 500 grams.]

No. of fraction.	Steam temperature corrected.	Weight of fraction.	Total weight.	Percentage weight.	Specific gravity at 15° C.	Index of refraction at 15° C.
	° C.	Grams.	Grams.	Per cent.	Sp. g.	N D.
1.....	94.9	51.50	51.50	10.3	0.8662	1.4692
2.....	94.9	53.09	104.59	20.9	.8659	1.4694
3.....	94.9	52.75	157.34	31.5	.8659	1.4697
4.....	94.9	50.92	208.26	41.7	.8660	1.4697
5.....	95.1	58.01	266.27	53.3	.8659	1.4697
6.....	95.1	54.61	320.88	64.2	.8658	1.4697
7.....	95.1	48.35	369.23	73.9	.8660	1.4699
8.....	95.7	47.03	416.26	83.3	.8662	1.4707
9.....	96.4	44.42	460.68	92.1	.8685	1.4722
10.....	98.0	a 30.00	490.68	98.1	.8756	1.4757

a About.

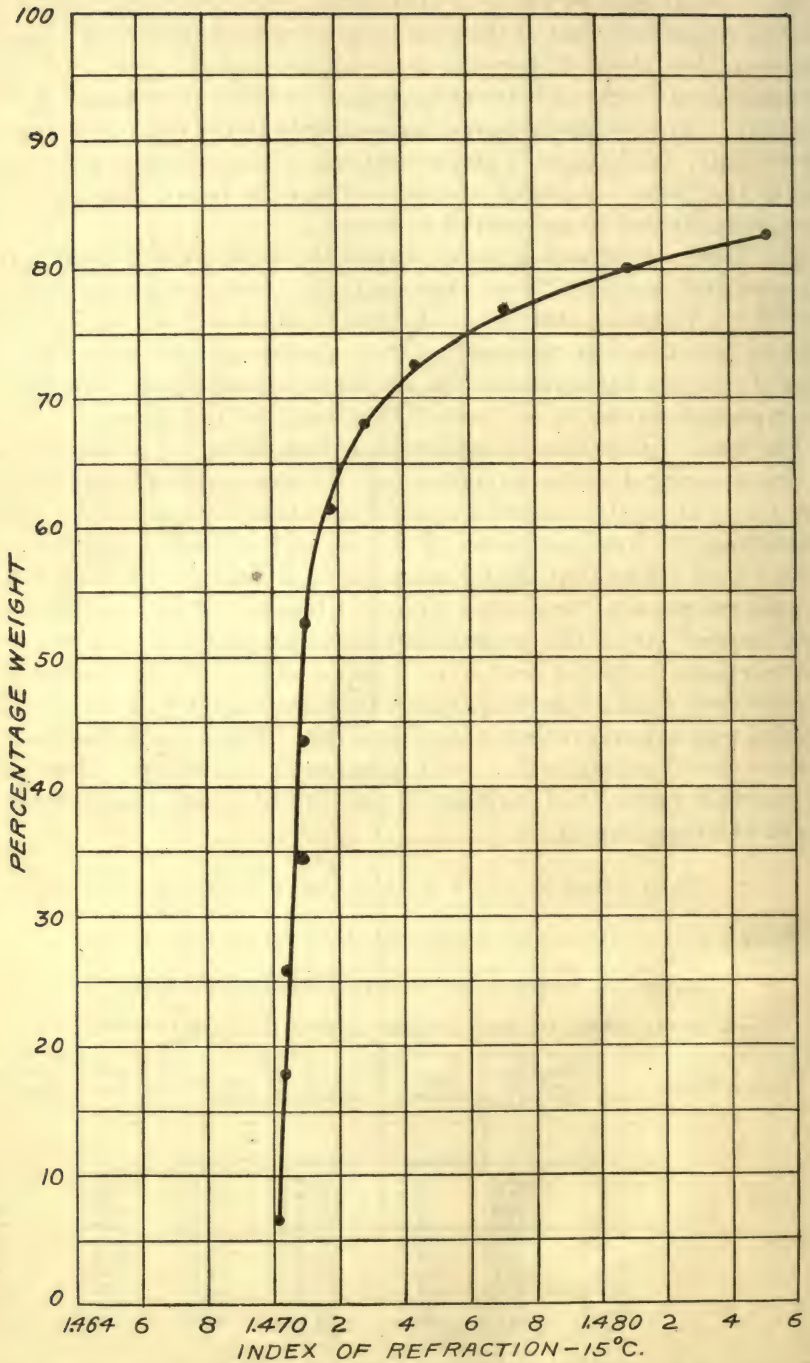


FIG. 10.—Percentage weight-index of refraction curve for a crude steam-distillation turpentine.
[Cir. 152]

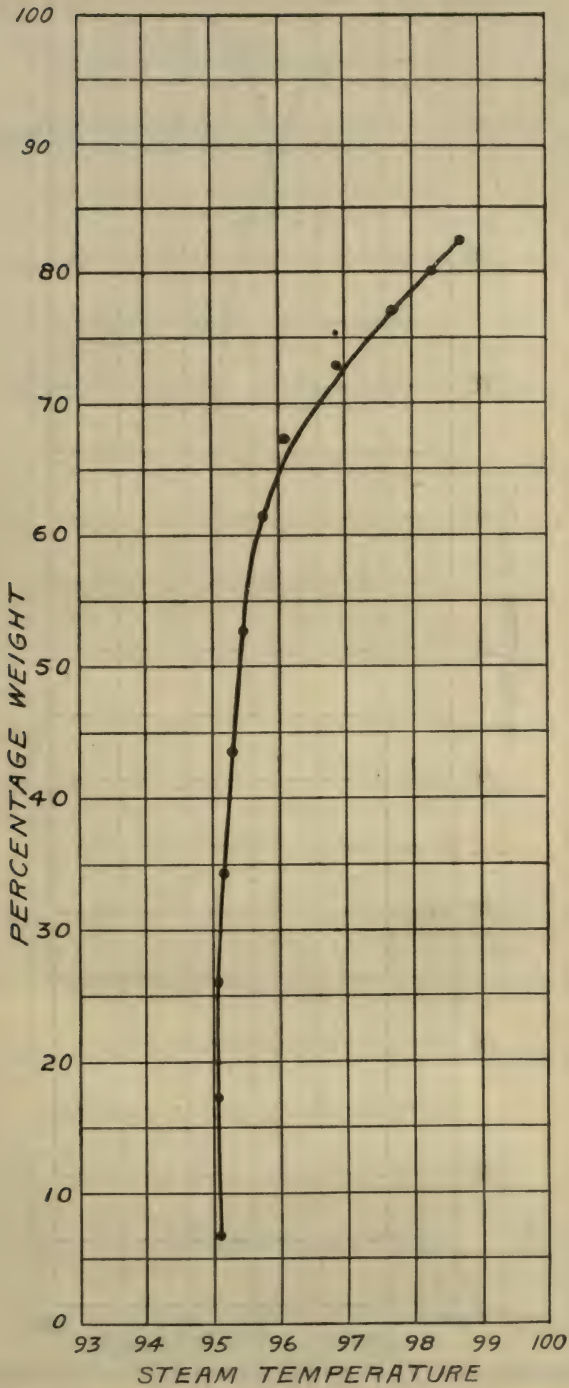


FIG. 11.—Percentage weight-steam temperature curve for a crude steam-distillation turpentine.
[Cir. 152]

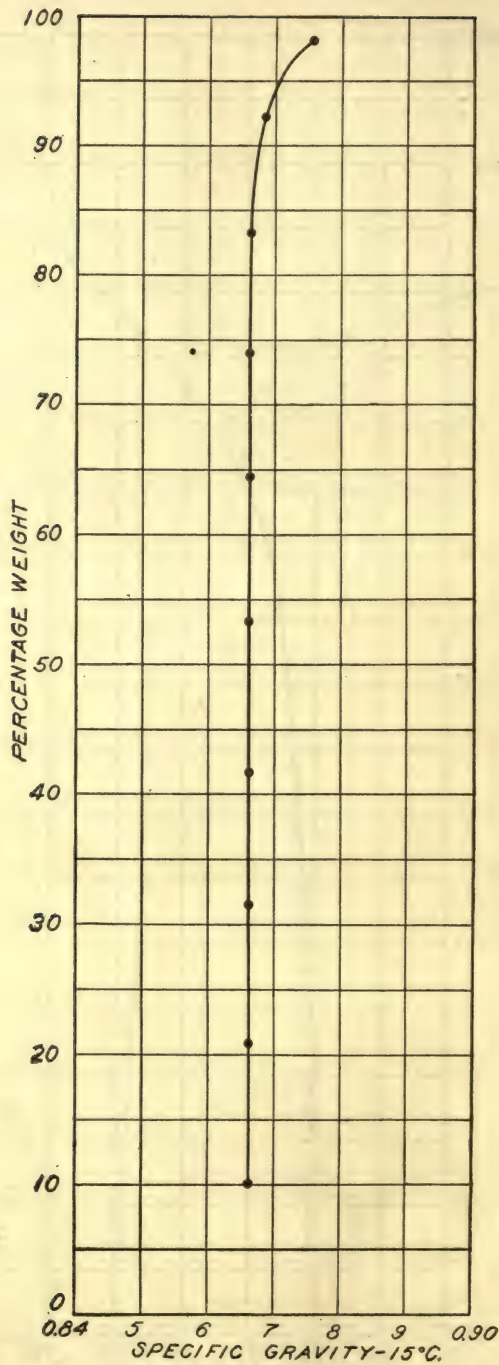


FIG. 12.—Percentage weight-specific gravity curve for a refined steam-distillation turpentine.

[Cir. 152]

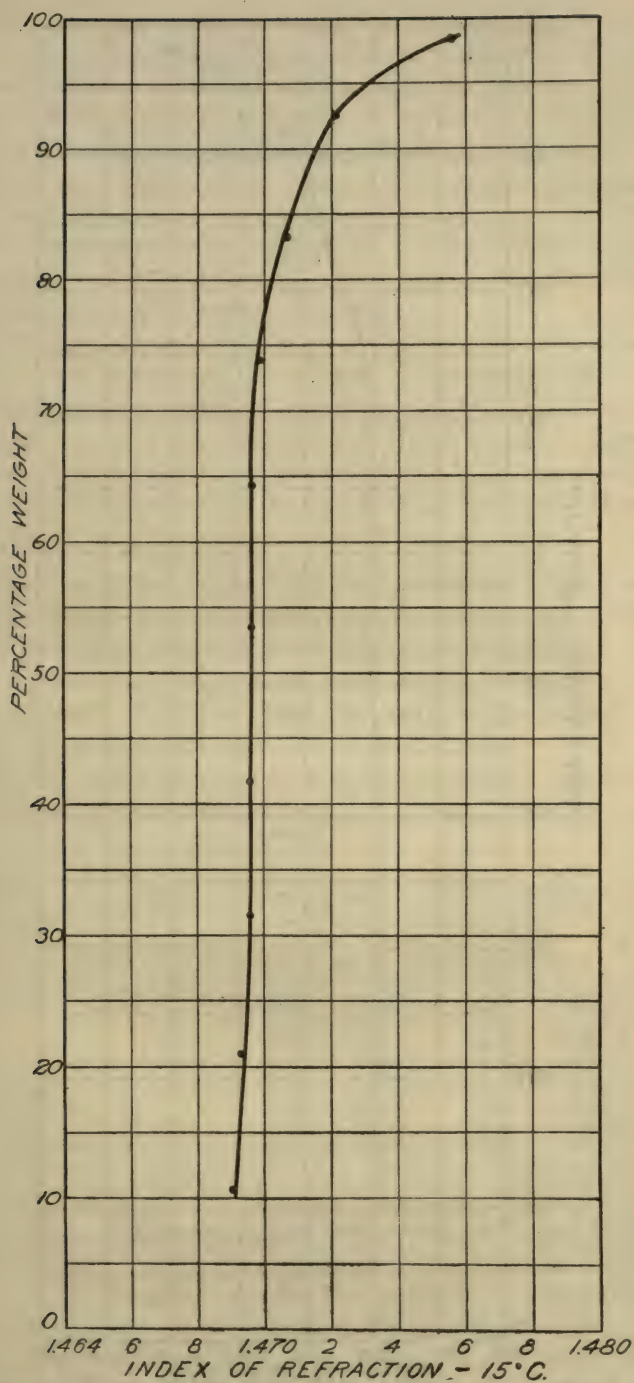


FIG. 13.—Percentage weight-index of refraction curve for a refined steam-distillation turpentine.
[Cir. 152]

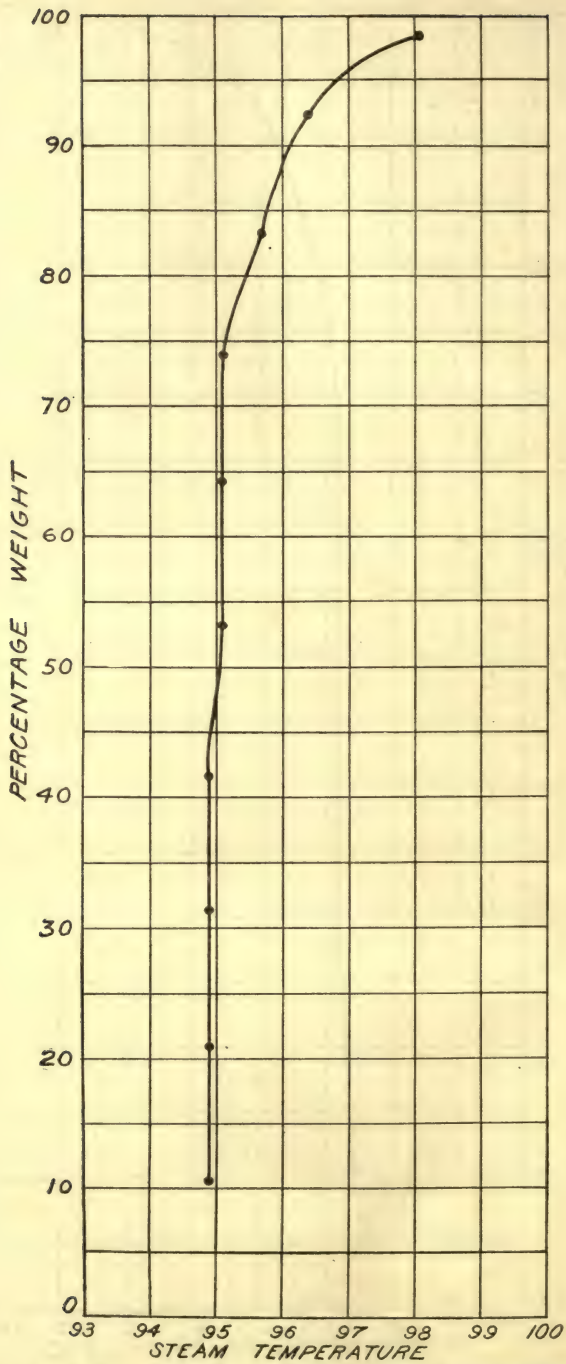


FIG. 14.—Percentage weight-steam temperature curve for a refined steam-distillation turpentine.
[Cir. 152]

The specific gravity curve is shown in figure 12. The specific gravity of the first 92 per cent of the distillate varied within the limits of 0.8662 and 0.8685. This is almost exactly the difference found in figure 1. The vertical part of the curve is very straight, and change of direction does not take place until nearly all the oil has distilled. This indicates, then, that the product was of high grade, and consisted chiefly of a pure light oil.

The index of refraction curve, figure 13, is very similar to that for the gum turpentine shown in figure 7. This change over 92 per cent is from 1.4692 to 1.4722, a difference of 0.0032.

The steam temperature curve, figure 14, further shows marked constancy over a large portion of the oil, and although not quite so perpendicular as the curve shown in figure 8, still within 1.5° variation in temperature the same per cent of oil distilled, namely, 92 per cent. The inference, then, is that this turpentine is of a high degree of purity, being practically the equal of the gum turpentine previously described.

TURPENTINE No. 22—A CRUDE DESTRUCTIVE-DISTILLATION TURPENTINE.

The possibilities of this method are further shown in the results of an analysis of crude turpentine produced by destructive distillation of pine wood. Table 4 presents the data from this analysis. Figures 15, 16, and 17 are the curves made from this table for the specific gravity, the index of refraction, and the steam temperature, respectively.

TABLE 4.—*Turpentine No. 22—A crude destructive-distillation turpentine.*

[Specific gravity, 0.9231 at 15°; index of refraction, too dark for a determination; weight taken, 500 grams.]

No. of fraction.	Steam temperature corrected.	Weight of fraction.	Total weight.	Percentage weight.	Specific gravity at 15° C.	Index of refraction at 15° C.
	° C.	Grams.	Grams.	Per cent.	Sp. g.	N D.
1.....	94.1	10.20	10.20	2.0		1.4584
2.....	94.7	13.66	23.86	4.8	0.8674	1.4691
3.....	95.1	26.71	50.57	10.1	.8656	1.4703
4.....	95.3	36.03	86.60	17.3	.8644	1.4719
5.....	96.1	46.23	132.83	26.6	.8631	1.4737
6.....	96.5	42.42	175.25	35.1	.8620	1.4763
7.....	96.9	39.71	214.96	43.0	.8603	1.4791
8.....	97.6	34.02	248.98	49.8	.8628	1.4852
9.....		24.23	273.21	54.6	.8728	1.4910
10.....	98.5	18.74	291.95	58.4	.8904	1.4962
11.....	98.7	15.33	307.28	61.5	.8933	1.4993

On reference to figure 15 the oil seems fairly uniform over 50 per cent of its composition, with a variation between 0.8678 and 0.8600, or 0.0078. The specific gravity, however, decreases with the distillation, whereas that of all the other samples has increased. This indicates a very different composition. The irregular form of the index of refraction and steam temperature curves (figs. 16 and 17) shows that the oil was one of very complex composition.

The odor, both of the sample and the fractions, was strong and disagreeable, while the color varied from a yellow to a red.

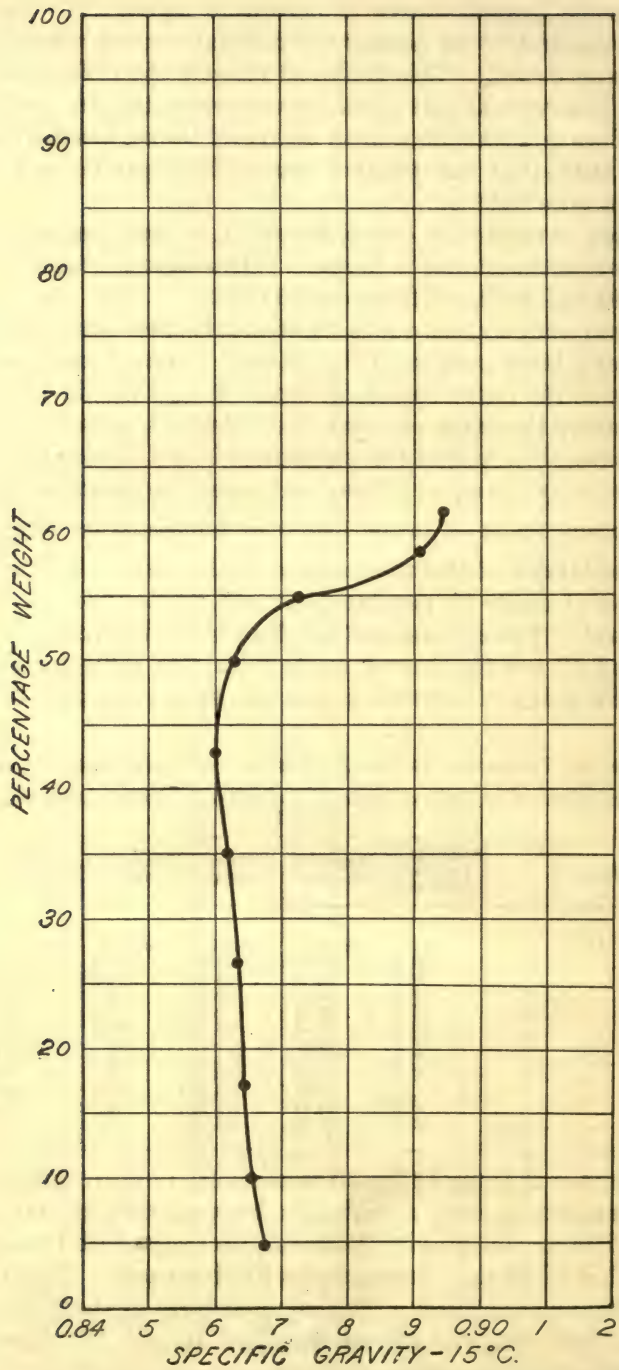


FIG. 15.—Percentage weight-specific gravity curve for a crude destructive-distillation turpentine.
[Cir. 152]

It is perfectly possible, then, to distinguish a complex oil by the irregularities in the shape of the curve, and when several oils of widely varying properties are present in any mixture this method of analysis will show their presence.

CONCLUSIONS.

This article has outlined a method of analysis by fractional distillation with steam, and has shown the results of the application of this method to four turpentines of different kinds.

In discussing the results three physical properties have been given emphasis—specific gravity, index of refraction, and temperature of

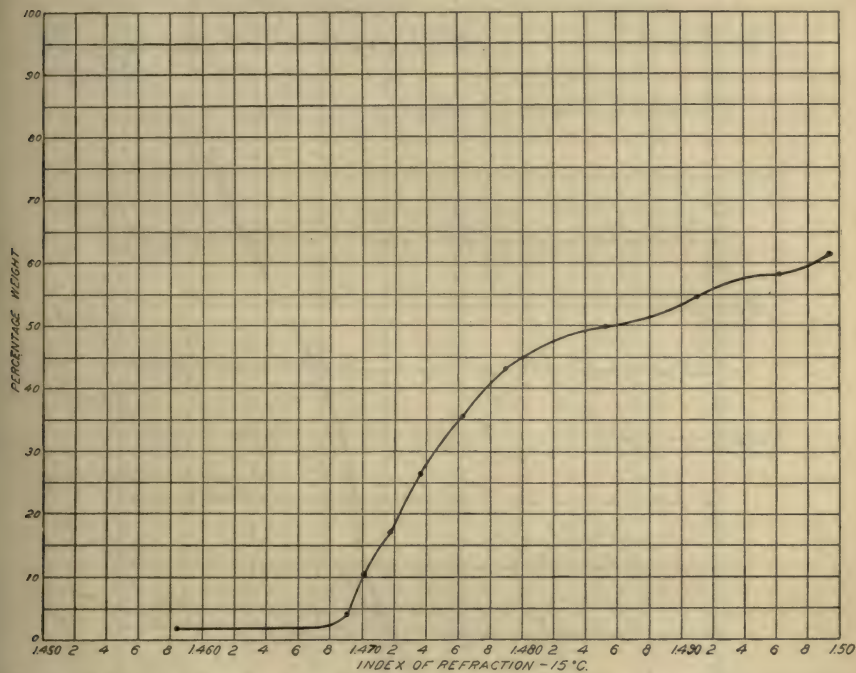


FIG. 16.—Percentage weight-index of refraction curve for a crude destructive-distillation turpentine.

distillation with steam. These three properties were chosen because they lend themselves to rapid determination and clear interpretation. The odor and color were also found to be of value in the final conclusions. The basis for the judgment of an oil has been the combined interpretations from the various physical properties as indicated in the shape of the curves, and no one physical property has been assumed to be all-conclusive. The interpretations from the various physical properties have been seen, however, to harmonize very well.

Certain advantages of this method seem worthy of mention.

(1) By introducing the principle of dephlegmation the separation of the constituents is made much more complete.

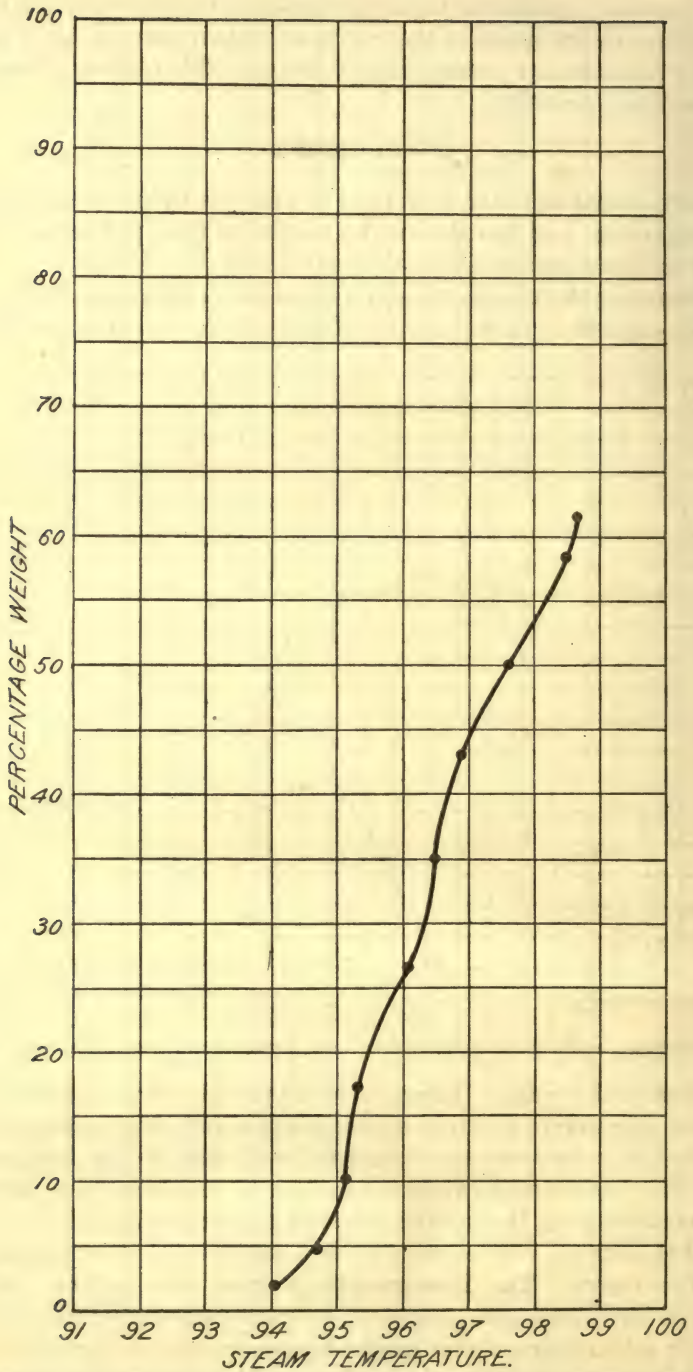


FIG. 17.—Percentage weight-steam temperature curve for a crude destructive-distillation turpentine.
[Cir. 152]

(2) The graphical method of expressing the data gives a ready means of interpreting the results and comparing various turpentines.

(3) The large amount of data obtained makes the results very reliable.

(4) The low temperature at which the distillation is carried out reduces the danger of alteration of the sample during the analysis.

(5) The distillation is similar to certain technical distilling processes which have been found to give excellent results in practice, and suggests the possible results of applying such methods to the refining of turpentines.

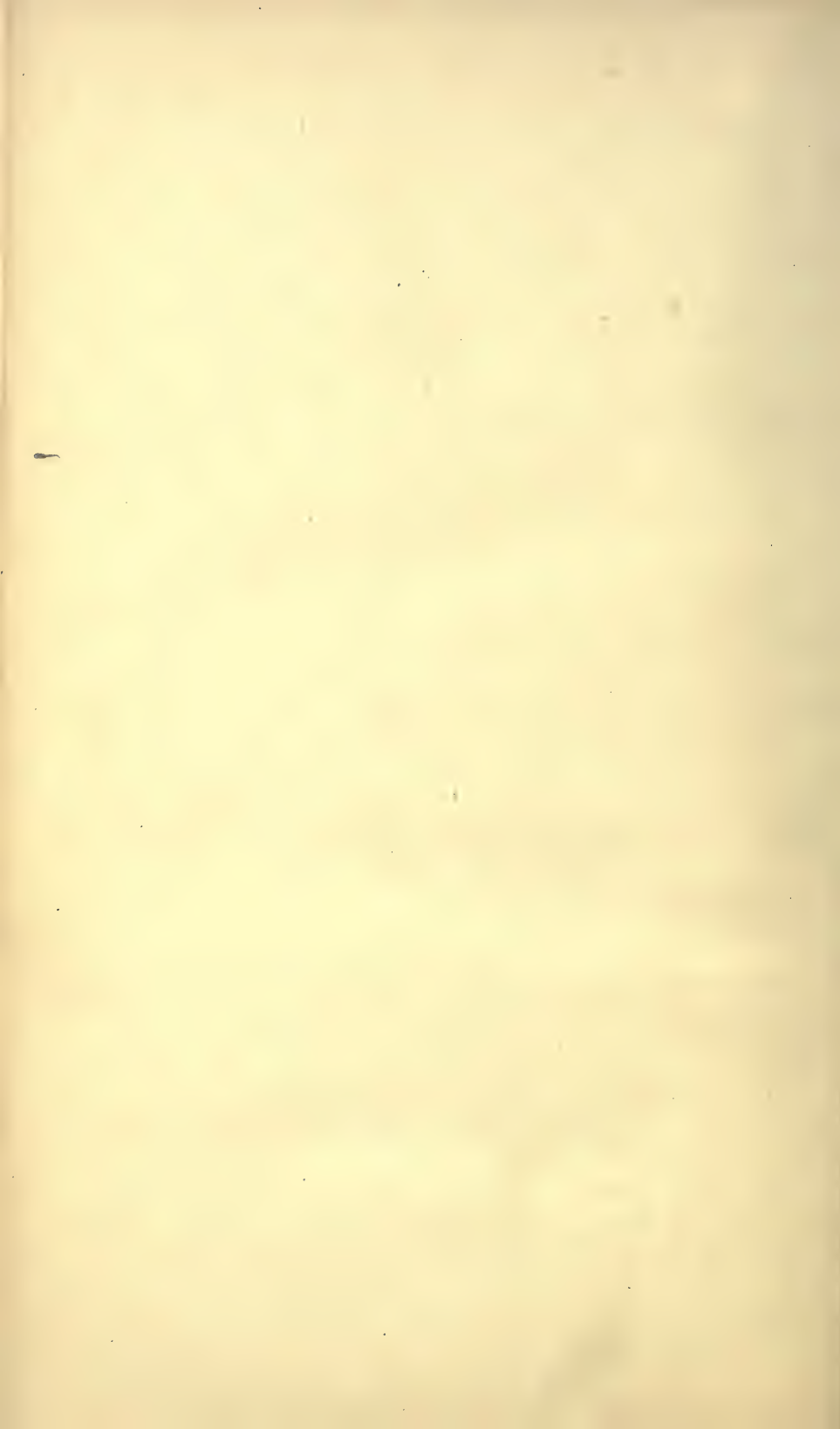
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JAMES WILSON, *Secretary*.

WASHINGTON, D. C., *May 8, 1908.*

[Cir. 152]

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HENRY S. GRAVES, Forester.

FUSTIC WOOD:

ITS SUBSTITUTES AND ADULTERANTS.

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GEORGE B. SUDWORTH
University of California

DENDROLOGIST,

AND

CLAYTON D. MELL,

ASSISTANT DENDROLOGIST.



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FUSTIC WOOD: ITS SUBSTITUTES AND ADULTERANTS.

Buyers of fustic wood are often in doubt regarding the genuineness of some of the material put upon the market as true fustic. A microscopic study of a large number of samples of so-called fustic wood submitted to the Forest Service showed that several different woods are substituted for true fustic, or are used to adulterate it in a chipped or ground condition. The purpose of this circular therefore is to point out the distinguishing characteristics of genuine fustic wood and of its substitutes, in order that consumers may readily distinguish the real from the spurious article.

TRUE FUSTIC WOOD.

True fustic wood which yields the valuable yellow, brown, and green dyestuffs, comes from the fustic tree (*Chlorophora tinctoria* Gaud. = *Maclura tinctoria* D. Don = *Morus tinctoria* L), a native of the West Indies and tropical America. The tree is also called old fustic, fustic mulberry, Cuba wood, yellowwood, and mora. It should not be confused, however, with the true mora (*Dimorphandra mora* B. and H. = *Mora excelsa* Benth.) of British Guiana, Venezuela, and part of Central America, the wood of which is readily distinguished from that of freshly cut fustic by its reddish-brown color. The fustic tree attains a height of from 25 to 50 feet, and a diameter of 2 feet or more. The nearly white sapwood is very thin, and the greater portion of the tree therefore is heartwood, the only part put to commercial use. Freshly cut heartwood is light yellow, but after exposure to air and light becomes a yellowish brown. A cubic foot of seasoned wood weighs approximately 50 pounds.

Most of the fustic wood used in Europe comes from South America, while the bulk of that consumed in the United States comes from Mexico and the British West Indies. The amount entered at United States ports for immediate consumption during the year ending June 30, 1909, together with the withdrawals from warehouses for later use, was 2,466 tons, valued at \$34,752. The average value per ton for 1909 was \$14.09, an increase over the previous year of \$1.99.

Fustic wood is usually imported in sticks from 2 to 4 feet long and from 3 to 8 inches in diameter, but appears also in the form of chips,

powder, aqueous extract, and paste or lake. The greater part of the supply, however, comes on the market in either chips or large pieces. While it is possible with a microscope to detect fibers of other woods mixed with ground fustic, it is often impossible, from the fibers alone, to identify them, because the distinguishing characters of the fibers



FIG. 1.—Transverse section of fustic wood (*Chlorophora tinctoria*). Magnified 50 diameters. Vessels are the large circular or elliptical openings scattered throughout the transverse section. Wood fibers comprise the broad, dark, dense, bands extending horizontally across the illustration. Wood-parenchyma fibers form the lighter, narrow, horizontal bands which extend across the picture; this tissue is seen to extend more or less around all vessels. Pith rays are the somewhat indistinct, narrow lines extending from the top to the bottom of the illustration.

have been obliterated by grinding. It is comparatively easy, however, to identify the different woods before they are powdered or ground into pulp.

SUBSTITUTES FOR FUSTIC WOOD.

The woods most commonly substituted for fustic are of the smoke-tree, osage orange, and also several species of prickly ash (*Xanthoxylum*), the latter being occasionally sold under the names prickly yellowwood or yellowwood. Since true fustic is becoming scarce,

substitution of other woods is likely to be practiced even more extensively in the future than at present. Manufacturers of coloring extracts derived from fustic are now forced to compete with the manufacturers of extracts from similar dyewoods prepared by the synthetic process, and the small margin of profit on the genuine extract increases the temptation to substitute cheaper woods.

SMOKE-TREE.

(*Cotinus cotinus* (L.) Sarg.)

The wood of the smoke tree, an Old World species, was formerly sold as the young branches of old fustic, and therefore was often

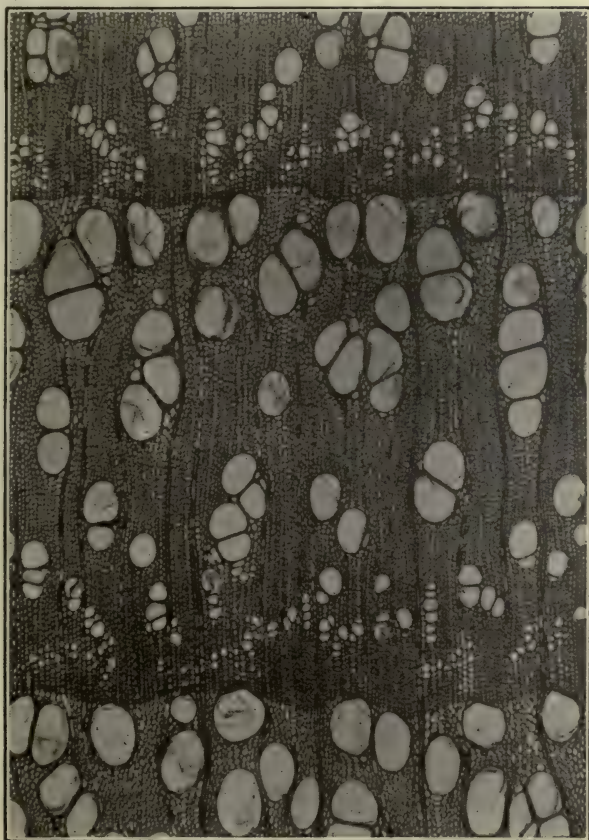


FIG. 2.—Transverse section of smoke-tree wood (*Cotinus cotinus*). Magnified 50 diameters.

called young fustic. It is known also as Aaron's beard, false fringe tree, Venetian sumach, wigtree, young mastic, and Hungarian fustic. It is a large shrub or small tree, yielding sticks from 4 to 6 feet long and seldom over 4 inches in diameter. The heartwood, which is golden yellow, lustrous, and hard, gives a yellow dye for wool and leather. Most of this wood comes from Greece and other Mediterranean countries. The available supply, however, is limited.

A comparison of transverse sections of smoke-tree and fustic wood (figs. 1 and 2) clearly distinguishes one from the other. Instead of being diffuse porous¹ with indistinct annual layers of growth, as old fustic, smoke-tree wood is ring porous² and has distinct annual layers of growth with a preponderance of large pores in the early wood (inner part of the layer), which gradually diminish in number and size toward the outside of the layer. In smoke-tree wood the vessels and tracheids have large simple pits,³ side by side, together with transitional forms merging into true bordered pits,³ but never bordered pits alone. The markings on the walls of vessels in true fustic consist of round bordered pits only. The simple pits in the vessels of smoke-tree are in most cases elliptical, lying parallel to one another, and transversely to the longitudinal axis of the elements, so that collectively they resemble the scalariform perforations of vessels. The wood fibers of the smoke-tree vary from 0.60 to 0.94 millimeter⁴ in length, with an average of 0.78 millimeter, about 0.15 millimeter shorter than those of fustic.

OSAGE ORANGE.

(*Toxylon pomiferum* Raf.)

Osage orange is known also as osage, bois d'arc, bodock, bowwood, yellowwood, and hedge tree, and is native to the rich bottom lands of southern Arkansas and westward to Oklahoma and Texas. It has been planted extensively throughout the eastern and central United States, and is largely cultivated for hedges. By some it is claimed to be superior to fustic as a dyewood. The wood, which is yellowish, hard, heavy, and strong, is distinctly ring-porous and may readily be distinguished from that of smoke tree and true fustic by the conspicuous tangential bands of wood-parenchyma tissue near the outer boundaries of the layers of growth (fig. 3). The early wood begins with a more or less broad zone of greatly flattened wood-parenchyma fibers, among which are scattered large pores. The pores toward the outside of the annual ring of growth are very small and are within the tangential bands of wood-parenchyma fibers that

¹ In a diffuse-porous wood the pores are approximately the same size throughout the annual layers of growth and quite evenly distributed; this results in a wood of homogeneous structure, such as is shown in figures 1 and 4.

² In a ring-porous wood the pores are largest and most numerous in that portion of the annual layer of growth first formed; as a result, the annual layers are sharply defined, as seen in figures 2 and 3.

³ Pits are unthickened portions in the walls of wood elements. They are either round, elliptical, or slit-like, and are further divided into simple and bordered pits. Whether a pit is simple or bordered is determined by the character of the canals which extend from the middle lamella, or common wall of two adjacent elements, to the cavity of the cell. When a transverse or longitudinal section is prepared, a so-called profile view of pits will be seen, and if the walls of the canal are nearly parallel or diverge only slightly toward the common wall, as shown in figure *a*, it is a simple pit. If the walls of the canal make a distinct angle just inside the pit opening, as shown in figure *b*, it is known as a bordered pit.

⁴ A millimeter is about one twenty-fifth of an inch.



are not radially flattened. The wood fibers have thick walls, small cavities, and vary in length from 0.714 to 1.008 millimeters, with an average length of 0.865 millimeter, which is intermediate between smoke-tree and true fustic. The wood of this species (fig. 2) presents an entirely different arrangement of structural elements from that of

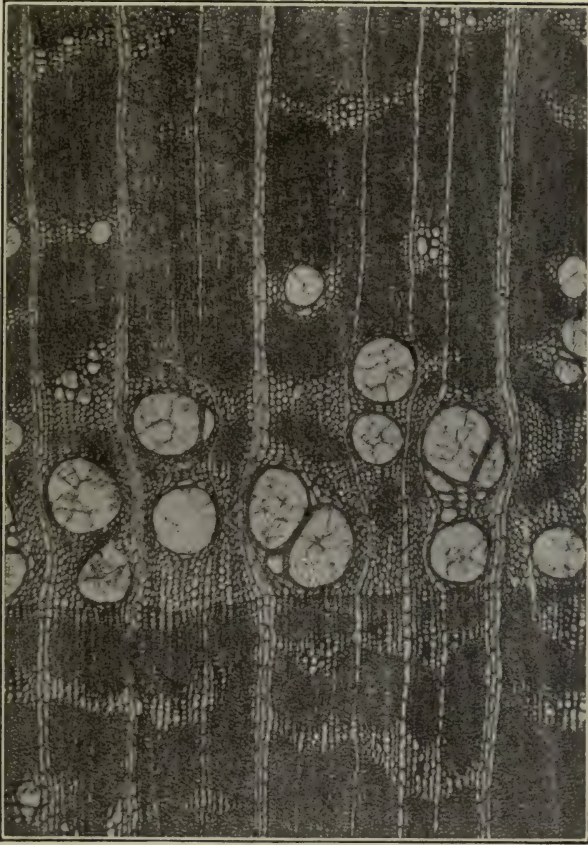


FIG. 3.—Transverse section of osage orange (*Toxylon pomiferum*). Magnified 50 diameters.

either smoke-tree or true fustic, and there should be no difficulty in distinguishing the three species if smooth transverse cuts are viewed with a hand lens magnifying 6 or 8 diameters.

SOUTHERN PRICKLY ASH.

(*Xanthoxylum clava-herculis* Linn.).

Besides southern prickly ash, which ranges from southern Virginia to Florida and westward to Texas, the woods of the following species of semitropical Florida and the West Indies have appeared as substitutes for fustic: Espino (*Xanthoxylum acuminatum* Swartz), yellowwood (*X. ochroxylum* D. C.), satinwood (*X. cribrosum* Spreng.),

and wild lime (*X. fagara* Linn. Sarg.). In the West Indies these woods are known by the general name of espino or espino rubial. Elsewhere they are known as box, yellow thorn, or yellow Hercules club. They furnish a hard, light yellowish wood, sometimes nearly white, but which becomes yellowish when exposed to light. The structural characters of the woods of these species are so similar that the southern prickly ash may be taken as typical of all.

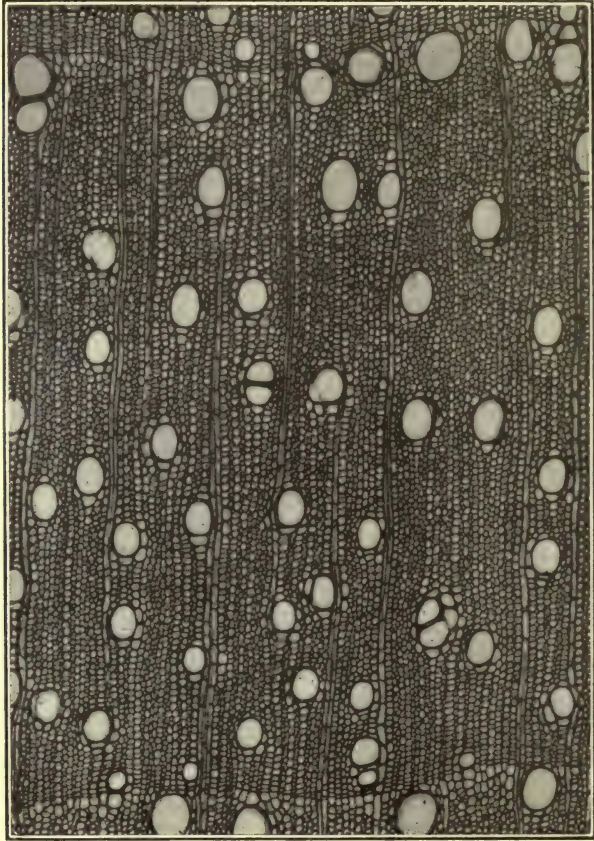


FIG. 4.—Transverse section of southern prickly ash wood (*Xanthoxylum clavaherculis*.) Magnified 50 diameters.

The wood of southern prickly ash is moderately heavy, hard, close grained, with yellowish-brown heartwood and very thin yellow sapwood. The annual rings of growth can be distinguished with the naked eye, especially in samples from the temperate Zone, but they are less distinct in specimens from Cuba or extreme southern Florida. The early wood is marked by a single line of pores or vessels which vary from 0.1 to 0.15 millimeter in diameter. These vessels are usually somewhat larger than those formed later. Vessels in the

late wood vary from 0.03 to 0.1 millimeter and are evenly distributed throughout the annual ring of growth; occasionally they are arranged radially in groups of from two to four. The perforations at the ends of the vessel segments resemble large simple pits. The side walls of vessels have bordered pits where they are in contact with the wood-parenchyma fibers or pith ray cells. The wood fibers which form the bulk of the secondary wood have simple pits and vary in length from 0.630 to 1.302 millimeters, with an average length of 0.939 millimeter. Wood-parenchyma fibers are not very abundant, occurring sparingly only near vessels and invariably contain small solitary crystals in each cell. The pith rays are very narrow, varying from one to three, but rarely more than two rows of cells in width. (Fig. 4.)

IDENTIFICATION OF FUSTIC WOOD AND ITS SUBSTITUTES.

The distinguishing characters of nearly all woods are more readily observed in transverse than in longitudinal sections, and for this reason the illustrations in this circular show magnified transverse sections of the wood of fustic, smoke-tree, osage orange, and southern prickly ash (figs. 1, 2, 3, and 4). It is chiefly upon the characters to be seen in these sections with an ordinary pocket lens that the following key is based:

A. Layers of growth indistinct (diffuse porous).¹

1. Tangential (wavy) bands of wood-parenchyma fibers² conspicuous, and containing numerous small pores filled with tyloses.

(a) Wood light yellow, retaining color indefinitely, moderately hard and heavy (about 43 pounds per cubic foot). *Chlorophora tinctoria* (fig. 1).

B. Layers of growth distinct (ring-porous).³

1. Tangential bands of wood parenchyma in the late wood more or less inconspicuous and interrupted.

(a) Wood orange yellow (when freshly cut), turning brown with exposure, hard and heavy (48.2 pounds per cubic foot). The large pores in early wood are completely filled with tyloses.

Toxylon pomiferum (fig. 2).

2. Tangential bands of wood-parenchyma fibers entirely wanting.

(a) Wood brownish yellow, moderately hard, and of lighter weight (about 40 pounds per cubic foot) than the preceding. Pores in early wood large and very numerous, gradually diminishing in size and number in the late wood..... *Cotinus cotinus* (fig. 3).

(b) Wood pale yellow, slightly tinged with red, moderately hard, and of lighter weight (31.51 pounds per cubic foot) than the preceding. Pores in early wood smaller than in the preceding, forming a single interrupted row, abruptly diminishing to a slightly smaller size and evenly distributed in the late wood, solitary or in groups of two to four *Xanthoxylum clavaherculis*.

¹ See footnote 1, p. 8.

² This tissue appears as fine lines or bands, usually wavy and often interrupted, running in the same direction as the annual layers of growth, and is seen in figure 1.

³ See footnote 2, p. 8.

ANATOMICAL CHARACTERS OF FUSTIC WOOD.

A full description of the microscopic structure of fustic wood is presented here for the help it may give in distinguishing true fustic from possible new, and at present unknown, substitutes whose gross characters may not, as in the case of the osage orange, smoke-tree, and prickly ash, suffice for identification.

Fustic wood does not exhibit true annual rings of growth. The alternating bands of wood parenchyma and wood fibers are sometimes mistaken for such, the softer tissue appearing as early wood and the dense tissue as late wood. This error may be avoided, however, by remembering that early wood is never composed of tissue wholly different from that of the late wood. Moreover, the absence of an intermediate zone showing gradual transition from early to late wood is further evidence that these are not annual rings of growth.

A radial cut of true fustic wood made with a sharp knife shows alternating tangential bands of different tissues, which are quite conspicuous when viewed with a hand magnifier. Their prominence is due not so much to the difference in color as to the variation in density and character of the elements composing them. The main tissue, consisting of very hard, thick-walled wood fibers, forms dense undulating bands alternating with layers of less dense tissue composed of thin-walled wood-parenchyma fibers, and known as wood-parenchyma bands. These are seldom continuous around the stem or branch, but are very irregular, often quite short or meeting with the next inner or outer bands of similar tissue. They are from one-third to one-half the width of the bands of wood fibers alternating with them. The bands of wood fibers vary from 2 to 6 cells in width, except in regions of pores, where they are often from 10 to 12 cells wide. The pores lie within the bands of wood parenchyma and are wholly surrounded by them.

Vessels (transverse section)¹ vary from 0.02 to 0.25 millimeter in diameter, are more or less evenly distributed throughout the wood, and are easily seen with the aid of a pocket lens magnifying from 4 to 6 diameters. The walls are usually quite thick, especially on the side bordering other vessels. The vessels are wholly surrounded by wood-parenchyma fibers, and usually are much greater in diameter than the average width of the bands, which are thus rendered undulating. They are regularly distributed either singly, or less often in radial groups of 2 to 4, and are invariably filled with a yellowish mass called tyloses, which forms a network of cross walls within the vessel cavities. The vessel segments, which may, with aid of the microscope, be seen in longitudinal sections or in macerated material, are exceptionally short, seldom more than 0.5 millimeter long.

¹ See description (fig. 1).

The end cross walls are wholly absorbed, leaving an open passage connecting the abutting segments. These end walls are usually horizontal, though there are exceptions, especially in segments of the largest vessels, which have more or less oblique ends, each with an elongated pointed projection extending along the side of the next segment above and below.

The walls of the vessels have bordered pits. Where vessels are in contact with wood-parenchyma fibers or pith-ray cells the pits are arranged in groups, and although they are bordered, at least in the walls of the vessel, a slight transition to large simple pits occasionally occurs in the adjoining elements. The pit canals are round on the outside of the vessel walls, but they gradually widen out laterally on the inner walls of the vessels into slitlike canals which communicate one with another. The small vessels, especially those resembling tracheids, have markings on their walls similar to those of vessels; spiral-like markings, such as are found in osage orange, are nowhere present in this species. True tracheids have not been found in this wood; all elements with small cavities and bordered pits have simple perforations, at least at one end, and are thus to be classed as parts of vessels.

*Wood fibers*¹ are arranged radially in regular rows. They have exceedingly thick walls, the cavities being almost entirely obscured. In the region of large pores, which by virtue of their rapid growth exert considerable lateral pressure, the wood fibers and wood-parenchyma fibers are flattened radially; otherwise they are either circular, oval, or polygonal. Wood fibers vary in length from 0.65 to 1.18 millimeters, with an average of 0.939 millimeter. They are often less than 0.02 millimeter in diameter, and are long acuminate at both ends. The markings on the cell walls are indistinct, the pits being simple, minute, and arranged in a single row, chiefly on the radial walls.

*Wood-parenchyma fibers*¹ are arranged in tangential bands forming structures which contrast strongly with the bands of harder tissue bordering on the outside and inside. In transverse section these elements are often twice as wide as the wood fibers, or about 0.04 millimeter, their walls much thinner, and their cavities relatively larger and invariably filled with a parenchymatous substance easily soluble in alkali. Wood-parenchyma fibers bordering vessels are generally compressed either in radial or tangential direction; elsewhere they are circular or polygonal. They are somewhat shorter than wood fibers, and are composed of from 3 to 8 wood-parenchyma cells (more often 3 to 5) having horizontal or sometimes slightly oblique cross walls. The walls of these elements are marked on all sides by simple

¹ See description (fig. 1).

pits. Crystals of calcium oxalate are exceedingly abundant in wood-parenchyma fibers,¹ especially in cells bordering pith rays. These polygonal crystals occur in perpendicular rows varying from few to 8 or 10.

*Pith rays*² are regularly distributed throughout the wood. They are from 1 to 3 cells wide and from few to 30 or more cells high. They range in height from 0.2 to 0.7 millimeter. In a tangential section the pith-ray cells may be observed to vary considerably in size. Within the layer of denser tissue the rays are very narrow and the cells are invariably compressed laterally; within the bands of wood-parenchyma the pith rays are broader and the cells composing them larger and perfectly round in transverse sections. Ray cells bordering wood-parenchyma fibers occasionally contain crystals of calcium oxalate, but these crystals, very numerous in this wood, are found mainly in wood-parenchyma fibers. The individual cells of such fibers are cubic, and are usually much larger than the ordinary wood-parenchyma cells. The sculpture of the walls of the pith-ray cells is similar to that of the cells of the wood-parenchyma fibers.

¹ Parenchymatous elements containing crystal are known as idioblasts.

² See description (fig. 1).



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QUEBRACHO WOOD AND ITS SUBSTITUTES.

BY

CLAYTON D. MELL,
Assistant Dendrologist,

AND

WARREN D. BRUSH,
Expert.



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QUEBRACHO WOOD AND ITS SUBSTITUTES.

WOODS CALLED QUEBRACHO.

Quebracho (*Quebrachia lorentzii* Griseb.), a South American wood yielding a valuable extract much used in the United States for tanning high-grade leathers, is not the only wood known by that name. Two other and inferior species, *Aspidosperma quebracho-blanco* Schlecht and *Aspidosperma quebracho-colorado* Schlecht, are called white quebracho, or "quebracho blanco," and red quebracho, or "quebracho colorado," respectively. These two, however, belong to an entirely different family (*Apocynaceae*), including the common dogbane or Indian hemp, from the true quebracho, which is a member of the sumac family (*Anacardiaceae*). The name quebracho is derived from two Portuguese words, "quebrar," meaning break, and "hacha," ax, in allusion to the extreme hardness of the wood. At one time nearly every South American wood that quickly dulled an ax was called quebracho, but to-day the three woods mentioned are the only ones of commercial importance to which the name is applied.¹ This indiscriminate use of the name quebracho naturally has resulted in much confusion regarding the identity, distribution, and uses of these woods, and the purpose of this circular is to give the uses and distinguishing characteristics of each. Throughout, the true quebracho will be referred to simply as quebracho, while the two other species will be called white quebracho and red quebracho, respectively.

DISTRIBUTION AND SUPPLY OF QUEBRACHO.

Quebracho is found in Argentina, Bolivia, Brazil, Paraguay, and Uruguay. (See map.) It is distributed from the Andes eastward for approximately 500 miles, and from near the mouth of the Parana River northward for about 600 miles, thus growing over a territory of 300,000 square miles. A few writers claim that the Province of Santiago, Chile, once yielded quebracho, but nothing definite exists to show that it ever extended that far westward. Its commercial range is confined chiefly to northern Argentina, in the Provinces of Tucuman, Santiago del Estro, and Santa Fe, and the Territories of Formosa

¹ In Porto Rico a sapindaceous shrub (*Thouinia stricta* Radlk.), noted for its very hard wood, is called quebracho. In Cuba there is a "quebracho" tree (*Copaifera hymenaeifolia* Moric.), belonging to the bean family (*Leguminosae*). In Chile and Honduras the name quebracho is given to a species of *Acacia*, another member of the bean family. "Quebracho flojo" or "sombra de toro" (*Iodina rhombifolia* H. and A.) is a handsome evergreen tree of Argentina belonging to the sandalwood family.

and the Chaco, and to the Chaco of Paraguay. It is comparatively rare in Bolivia and Uruguay, and in the Provinces of Salta and Jujuy, Argentina.

At the present rate of consumption, the supply of quebracho appears to be practically inexhaustible. Owners of quebracho timber claim that an average acre yields approximately 2 tons of the wood. A very conservative estimate of the total possible yield from the areas where quebracho occurs in commercial quantity places it at 168,000,000 tons, while the present yearly consumption of the wood for all purposes is a little less than 1,000,000 tons. The consumption, however, because of the scattering way in which quebracho grows, entails the cutting over each year of about 500,000 acres.

The price of quebracho lands varies greatly. In Paraguay they bring, as a rule, from \$6,000 to \$7,000 per square league,¹ (approximately 4,633 acres), or about \$1.50 per acre, though some areas can be purchased for as little as from 15 to 25 cents per acre. In the Argentine Chaco, on the other hand, certain tracts near railroads or waterways sell for \$2 per acre or more.

THE QUEBRACHO TREE.

Quebracho never grows in pure stands, but is scattered through open forests composed of many species. Seldom, in fact, are more than four or five quebracho trees found on an acre. It usually attains a height of from 50 to 70 feet, and a diameter of from 2 to 4 feet. It is neither a mountain nor a river-bottom tree, but grows best in the somewhat elevated stretches between the water courses, or in other situations with sandy soil and moderate atmospheric and abundant soil moisture. Its growth during early life is very rapid, fence posts being obtained in 10 years, but in later life is very slow. Little is known, however, concerning its average rate of growth to maturity. So far it has been free from attack by insects.

QUEBRACHO WOOD.

The heartwood of quebracho is one of the hardest, heaviest, and, in the region of its occurrence, most durable woods known. Highly tempered tools are required to work even the young green wood, and old wood is almost as hard as metal. It weighs about 78 pounds per cubic foot, and has a specific gravity of from 1.2 to 1.4, sinking, of course, in water. Nine quebracho ties of the size used in Argentina weigh a ton. Its lasting qualities are such that, in Argentina, ties of the wood appear to remain indefinitely in a perfect state of preservation. Logs left in the woods have been found absolutely sound after 25 years. The wood contains a very large amount of tannin, which

¹ Daily Consular Report, January 11, 1911.



FIG. 1.—Distribution of the quebracho tree (*Quebrachia lorentzii*).

acts as a preservative, and to which its extraordinary durability has been ascribed.

The sapwood of quebracho, forming about one-third of the whole, is nearly white. The heartwood, when freshly cut, is dark cherry-red, but becomes much darker with age. It is fine-grained and takes a beautiful polish. When thoroughly seasoned it does not warp, nor is it likely to check during seasoning.

USES OF THE WOOD.

GENERAL.

Quebracho wood is one of the principal commercial products of South America. Though its qualities fit it for use in many ways, its value for tanning extract and crossties is so great that nearly the entire annual cut is put to one or the other of these uses. In Argentina, however, where it is commercially most important, quebracho is employed for more purposes, though not in greater quantity, than any other wood.

There, because it is proof against decay, it finds place in wharves, dry docks, and quays, and as fence posts, beams, and cross arms. It is also used for bridge and ship building, general construction, furniture, and interior finish. The timber is usually seasoned for from two to four years before being used.

CROSSTIES.

Quebracho has been adopted throughout its range as a standard crosstie timber, and is gradually replacing the cast-iron ties once common. In Argentina quebracho ties for broad-gauge roads are 9 feet long, 10 inches wide, and 3 inches thick, and sell for from \$2 to \$2.50 each. The wood is so hard that spikes can not be driven into it, and holes must be bored clear through a tie for bolts to fasten the rails. About 18 quebracho ties are got from the average acre, a yield which appears small when contrasted with the 40 to 60 first-class white oak or pine ties obtained from an acre of our native forest. The difference is compensated, however, when it is remembered that the life of a quebracho tie may be practically indefinite, while that of our best native ties is seldom greater than eight years. Practically all the quebracho cut for ties is used in South America. Quebracho ties have been tried experimentally in the United States, but were found to lack resiliency and to crack badly with frost. The expense of boring holes for bolts was also another item against them.

TANNIN EXTRACT.

The heartwood of quebracho yields from 20 to 24 per cent of tannin, the sapwood from 3 to 4 per cent, and the bark from 6 to 8 per cent. It is this remarkable production, varying in different parts of the

tree's range, which justifies the extensive use for tan extract of a wood so valuable for other purposes. The bark and sapwood are seldom utilized, a fact which seems to indicate unnecessary wastefulness, since the tannin content of the former is equal to that of the lower grades of our native tanbarks; while the content of the latter is but slightly below that of our native chestnut wood.

Besides tannin, quebracho wood contains considerable, though relatively small quantities of red, gambier-like coloring matter used as a filler. Quebracho extract does not contain enough of the nontannin materials to produce by itself well-nourished leathers, and is therefore mixed in the proportion of 1 to 4, by weight, with other tannin material, such as the extracts of mangrove and chestnut, which are stronger in nontannin substances.

When cut for tan extract the trees are cleaned of their bark, sapwood, and branches, and then brought to the factory, where they are reduced to shavings or small chips. These are boiled or steamed to release the tannin, and the resulting fluid extract is evaporated to a thick paste, which, when dry, goes to the market in sacks or barrels. The cost of cutting quebracho logs and bringing them to the factory is much greater than that of manufacturing the extract itself. At present logs are hauled chiefly by ox teams to the nearest railroad or boat landing, and from there shipped to the reducing mill. It is in Paraguay, where quebracho trees are very scattered, that the transportation of logs is most difficult and expensive. Little or no market exists for quebracho timber or extract there, and practically all of the wood is shipped to Buenos Aires, where it is either consumed for extract or exported.

Quebracho obtained from the Provinces of Santiago del Estero and Santa Fe, Argentina, is particularly rich in tannin, and there also the trees reach their largest size. It is chiefly in these Provinces that quebracho is being extensively lumbered, the output steadily increasing from year to year. In the Provinces of Salta and Jujuy, where quebracho is rare, the inhabitants prefer for tannin the bark of two trees, *Acacia cebil* Griseb. and *Piptadenia cebil* Griseb., locally known as *cebil*. These barks are exceedingly rich in tannin material, in the case of the latter, at least, containing from 19 to 25 per cent.

IMPORTATION OF QUEBRACHO INTO THE UNITED STATES.

Quebracho wood and extract are now the leading exports of Argentina and Paraguay. Since their introduction in 1888, imports of these into the United States have increased steadily. In 1910, 38 per cent of all the tannin material used in the United States was quebracho. Nearly all of this came from Argentina. Table 1 shows the quantity and value of material imported during the fiscal years 1907 to 1910, inclusive.¹

¹ Compiled from Bureau of Statistics Bulletins 76 and 90, Imports of Farm and Forest Products.

TABLE 1.—Imports of quebracho wood and extract into the United States, 1907–1910.

Year.	Wood.		Extract.	
	Tons.	Value.	Pounds.	Value.
1907.....	66,810	\$840,779	76,033,584	\$2,319,785
1908.....	48,871	612,971	79,186,787	2,260,364
1909.....	66,113	731,795	102,004,981	2,740,530
1910.....	80,210	1,058,647	95,183,073	3,021,902

Quebracho wood enters the United States free of duty. A duty of from one-half to three-fourths of a cent per pound, however, is levied on the extract, which, in the new (1909) tariff schedule, is divided into two classes, according to its density or the percentage of tannin it contains. If less in density than 28° Baumé¹ the duty is one-half cent; if greater it is three-fourths of a cent. Transportation from Argentina to the United States is so cheap that many importers bring in logs and extract the tannin in this country, thus saving the high import duty. The price of quebracho extract f. o. b. Montevideo or Buenos Ayres, the two principal exporting points, is from \$80 to \$85 per ton, and that of logs from \$14 to \$20 per ton.

SUBSTITUTES FOR QUEBRACHO.

Both of the other woods which go to the market as quebracho, the white and the red, yield a relatively low percentage of tannin, and it is therefore unlikely that they are knowingly substituted to any extent for the true quebracho. Often, however, they may unintentionally be substituted for quebracho, through similarity of names. Quebracho is frequently called quebracho colorado, and red quebracho is also called this, and often merely quebracho. White quebracho, too, goes under the latter name. While quebracho and red quebracho may resemble each other superficially, white quebracho is readily distinguished from either by its light color. White quebracho is commercially more important than the red, and in some parts of South America has been used as a substitute for quebracho. It yields a much lower percentage of tannin, but the tannin itself is not essentially different from that of quebracho.

WHITE QUEBRACHO (QUEBRACHO BLANCO).

White quebracho is common throughout the regions where quebracho grows. It is most abundant in the wetter parts of northern Argentina, in the Provinces of Cordova, Santa Fe, Catamarce, Santiago del Estero, the southern portion of Tucuman, and the Chaco. It is an evergreen tree, reaching a height of from 60 to 100 feet, a

¹ An arbitrary scale devised by Baumé. It may be reduced to specific gravities by the following formulas:

(a) For liquids heavier than water, sp. gr. = $\frac{144}{144 - X}$. (b) For liquids lighter than water, sp. gr. = $\frac{144}{134 + X}$. X in each case being the reading by Baumé's scale.

diameter of from 1 to 3 feet, and remarkable for its erect stem and wide spreading crown. It is usually less bulky than quebracho, and branches nearer the ground, features which help to distinguish it in the forest.

The wood is strong, hard, and very heavy (specific gravity from 0.88 to 1.1), very close-grained, yellowish white in color, with a pinkish or rosy tint. It is not very durable in contact with the soil, yet much in demand for fence posts, rafters, flooring, railroad trucks, cross-ties, cabinet work, turnery, hubs, spokes, and cogwheels. Its dense, uniform structure makes it suitable as a substitute for boxwood (*Buxus sempervirens* L.) in wood engraving. Unless very carefully seasoned, however, it is likely to warp and twist, and is said to be subject to insect attack.

RED QUEBRACHO (QUEBRACHO COLORADO).

Red quebracho is also abundant throughout quebracho's range. Its wood when freshly cut is red, but becomes dark brown with age. It is used for practically the same purposes as white quebracho. Although in general its structural characters are similar to those of the former, it is said to be more durable in contact with the soil. While red quebracho may at first sight be mistaken for quebracho, its structural characters distinguish it. Moreover, its commercial importance is relatively so small that it is seldom mixed with the other wood.

KEY FOR THE IDENTIFICATION OF QUEBRACHO WOOD AND ITS SUBSTITUTES.

As an aid in distinguishing the wood of quebracho from that of the two inferior species, a key is given, in which are included such gross characters of the woods as may be seen in a smooth transverse section with the aid of a hand lens. For the use of students, several of the minute characters, which can be seen only under the high power of a compound microscope, are also included. Plates I and II show magnified transverse sections of quebracho and white quebracho wood. The structure of red quebracho is so similar to that of white that an illustration of it is unnecessary.

Pores (vessels) small (0.03 to 0.09 millimeters), open, and regularly distributed; tangential bands of large pores usually alternating with bands of smaller pores. *Wood-parenchyma fibers* abundant, scattered throughout the wood, often arranged in short tangential lines. *Wood fibers* 0.9 to 1.5 millimeters long. *Heartwood* dark pink, hard, and heavy (about 62 pounds per cubic foot).

(*Aspidosperma quebracho-blanco* Schlecht.)

Similar in structure to above, but heartwood red, becoming dark brown with age.

(*Aspidosperma quebracho-colorado* Schlecht.)

Pores (vessels) larger (0.10 to 0.13 millimeters), closed (in the heartwood) and irregularly distributed, usually grouped in radial rows of from two to four. *Wood-parenchyma fibers* confined to proximity of vessels, often forming conspicuous groups. *Wood fibers* very small in diameter (0.5 to 1.2 millimeters long). All cells of the heartwood are crowded with a yellowish-brown substance. *Heartwood* dark cherry-red, very hard, and heavy (about 78 pounds per cubic foot).

(*Quebrachia lorentzii* Griseb.)

Approved:

JAMES WILSON,

Secretary of Agriculture.

WASHINGTON, D. C., *March 15, 1912.*





Fig. 1.—Transverse section of the wood of quebracho (*Quebrachia lorentzii*) showing one entire annual ring of growth (a. r.); v., vessel; w. f., wood fibers; w. p. f., wood parenchyma fibers; p. r., pith ray. Magnified 25 diameters.

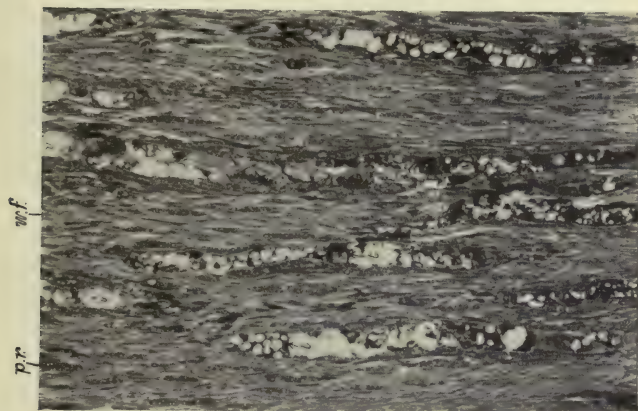


Fig. 2.—Tangential section of the wood of quebracho (*Quebrachia lorentzii*); v., vessel; p. r., pith ray; w. f., wood fibers; t., tyloses. Magnified 25 diameters.



Fig. 3.—Radial section of the wood of quebracho (*Quebrachia lorentzii*); v., vessels; w. f., wood fibers; p. r., pith rays; t., tyloses. Magnified 25 diameters.

THE WOOD OF QUEBRACHO.

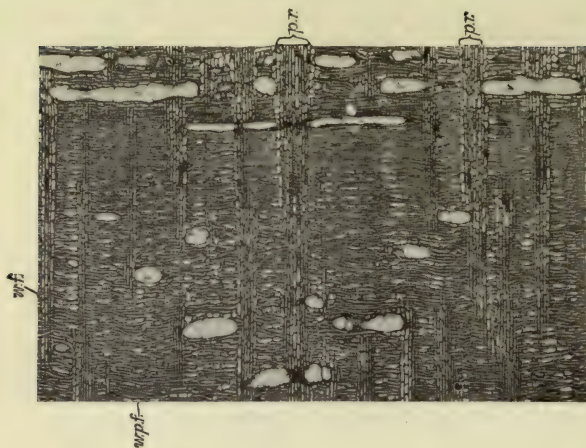


Fig. 1.—Transverse section of the wood of white quebracho (*Aspidosperma quebracho-blanco*) showing parts of two annual rings of growth; *v.*, vessels; *w. f.*, wood fibers; *w. p. f.*, wood-parenchyma fibers; *p. r.*, pith rays. Magnified 25 diameters.

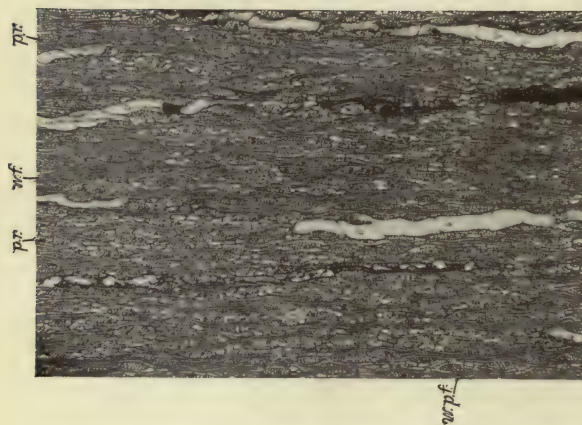


Fig. 2.—Tangential section of the wood of white quebracho (*Aspidosperma quebracho-blanco*); *v.*, vessel; *w. f.*, wood fiber; *w. p. f.*, wood-parenchyma fiber; *p. r.*, pith ray. Magnified 25 diameters.

THE WOOD OF WHITE QUEBRACHO.

Fig. 3.—Radial section of the wood of white quebracho (*Aspidosperma quebracho-blanco*); *v.*, vessel; *w. f.*, wood fibers; *w. p. f.*, wood-parenchyma fibers; *p. r.*, pith rays. Magnified 25 diameters.

UNITED STATES DEPARTMENT OF AGRICULTURE



BULLETIN No. 229

Contribution from the Forest Service
HENRY S. GRAVES, Forester



Washington, D. C.



July 28, 1915.

THE NAVAL STORES INDUSTRY.

By A. W. SCHORGER, *Chemist in Forest Products*, and H. S. BETTS, *Engineer in Forest Products, Forest Products Laboratory*.¹

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NEED FOR IMPROVED METHODS.

The business of producing naval stores is unique among American industries in one particular. Until recently there has been practically no change in methods since its first establishment. Those in vogue before the War of the Revolution are mainly the ones in force to-day. Very recently the cup method of turpentineing the trees has been taken up, but the wasteful box method is still largely used. Outside of this the average operator has been content to follow the methods of his predecessors, both in collecting the gum and in distilling it. With conditions in the industry as they are at present, when the supply of longleaf pine in North Carolina, South Carolina, and Georgia suitable for turpentineing is very nearly exhausted, and when the cost of operation everywhere is high in proportion to the returns,

¹ Maintained by the Forest Service in cooperation with the University of Wisconsin, Madison, Wis.
NOTE.—This bulletin is of interest to the producers and users of naval stores.

the need for improved methods, for attention to detail, is imperative if the industry is to have a future. Ways of collecting the gum which will give the maximum amount for the longest time with the least injury to the tree and methods of distillation which will insure turpentine and rosin of the best grade are things which every operator might well make the subject of study. Nowhere more than in the naval stores industry will close attention to detail coincide with successful operation.

This bulletin reviews the present status of the naval stores industry and the progress which has been made in improving the methods of collecting and distilling the gum.¹ Information is also presented on the supply of timber at present available for turpentine operations.

The publications listed in the latter part of this bulletin have been drawn upon in its preparation, and acknowledgement is made to the several authors.

HISTORY OF THE INDUSTRY IN THE UNITED STATES.

The earliest mention of turpentine and rosin seems to exist in a manuscript dated 1610, preserved in the Public Record Office, London, and entitled "Instructions for suche things as are to be sente from Virginia."²

"Hard pitche," "Tarre," "Turpentine," and "Rozen" are also mentioned in the "Booke of the Commodities of Virginia," issued presumably about the same time.

Pitch and tar were the chief products of the industry up to the middle of the eighteenth century. Their extensive use in the construction and maintenance of sailing vessels caused them to be called "naval stores," a term now applied to the turpentine and rosin industry, which has supplanted the production of tar and pitch.

The manufacture of turpentine and rosin in North Carolina was described by Schoepf in 1783-84. Pitch and tar, however, had been staple products since 1700. Norfolk was the great shipping point for Virginia and northeastern North Carolina.

The method of boxing the trees for collecting the crude gum was the same as that employed to-day, but the names of some of the operations have changed, such as "cornering" in place of "notching," and "virgin dip" in place of "pure dippings."

¹ The Bureau of Chemistry of the Department of Agriculture is now investigating problems connected with the distillation of turpentine, and has offered helpful suggestions in the case of this bulletin.

² Among the instructions is the following: "Pyne trees, or firre trees are to be wounded within a yarde of the grounde, or boare a hoal wth an agar the thirde pte into the tree, and lett yt runne into anye thinge that maye receyue the same, and that wth yssues owte wilbe Turpentyne worthe 18^e Tonne. When the tree beginneth to runne softelye yt is to be stopp'd vp agayne for preserueinge the tree."

"Pitche and tarre hath bene made there and we doubte not but wilbe agayne, and some sente for a sample, your owne tournes beinge firste served."

Previous to 1800 very little of the crude turpentine was distilled at the point of production. Tapping operations were conducted, so far as possible, along navigable streams and inlets. The crude turpentine was then shipped to Wilmington, Philadelphia, and New York, and there reshipped to England for distillation. Up to the year 1820 the production of turpentine and rosin was quite unimportant and limited to the demands of domestic industries. The rosin manufactured was worth very little, the price dropping to 25 cents a barrel and even lower, so that it could no longer be handled at a profit.

Copper stills were introduced in 1834, and greatly improved manufacturing conditions. Previously the distillations had been conducted in crude cast-iron retorts that gave very poor results, both as to quantity and quality of the products.

Up to the year 1838 the industry had not advanced south of the Cape Fear River, the belief being that the pines farther south would not flow sufficiently. This error was soon discovered through a few experiments, and the practically untouched belt of longleaf pine forest extending from the Carolinas to Texas was gradually invaded.

Increasing demands for turpentine in the varnish and paint industries; its utilization as a solvent for rubber and as an illuminant when mixed with alcohol, and the passage of the British free trade law of 1846 combined to stimulate production. In proportion as the demand for turpentine increased a greater amount of rosin was manufactured than could be utilized in the arts, and so went to waste.

The Civil War had a depressing effect upon the industry. Only about two-thirds of the number of establishments reported for 1860 were in operation in 1870.

STATISTICS OF PRODUCTION.

Except for the period between 1860 and 1870, the naval stores industry has had a steady growth, so far as the value of its products is concerned. The market prices are subject to great variation,¹ not only according to supply and demand, but also through manipulation on the exchange. Figure 1 shows the high and low prices by years since 1901 for turpentine and for two grades of rosin.

During the last five years the average annual production, in round numbers, has been 31,800,000 gallons of turpentine and 3,700,000 barrels of rosin. Over half of the products are exported, this country largely supplying the world. France, Austria, Spain, Portugal, India, Greece, and other countries also produce naval stores, but the amounts are relatively small, as may be seen from Tables 1 to 4, which show the exports and imports of turpentine and rosin by various countries from 1901 to 1912.

¹ During the season 1909-10 the lowest price of turpentine per gallon was 35½ cents, while during 1910-11 the high-water mark of \$1.07 was reached. The lowest quotation ever posted by the Savannah Board of Trade was on Sept. 4, 1896, when turpentine brought but 22 cents a gallon.

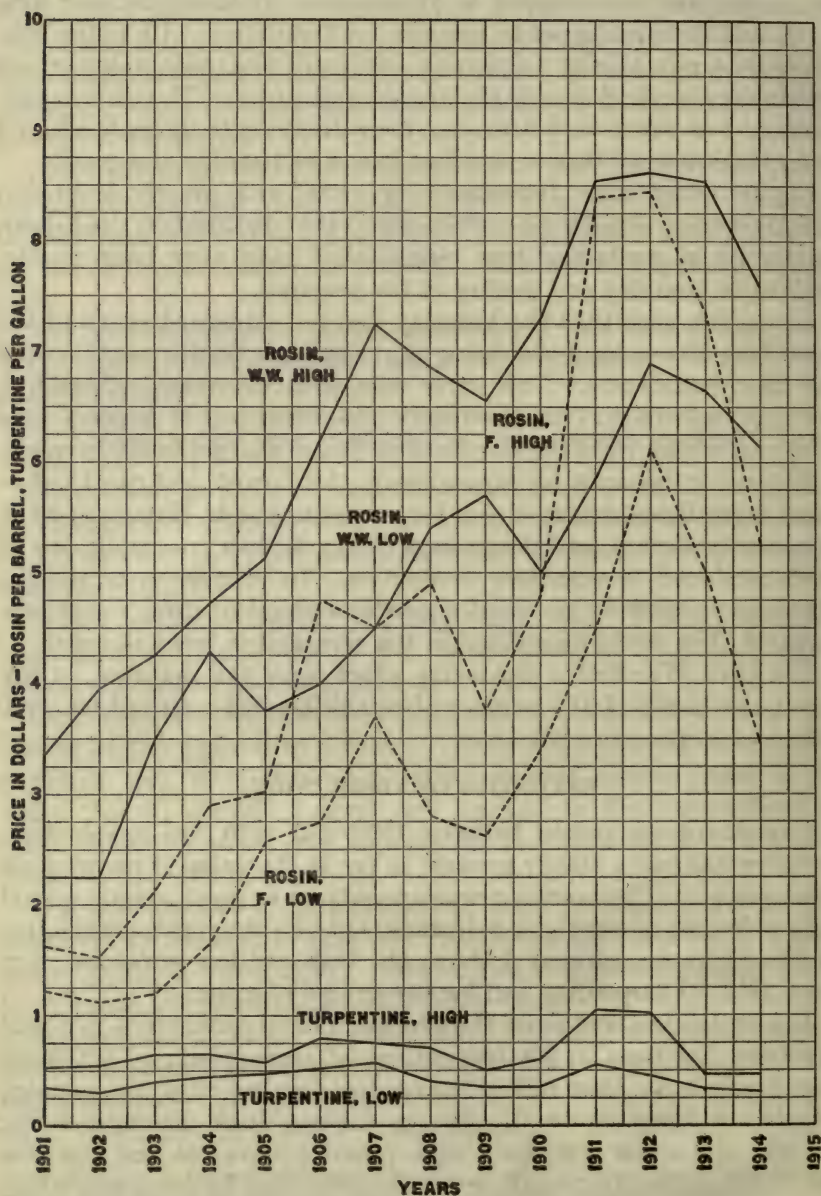


FIG. 1.—High and low prices by years since 1901 for turpentine and two grades of rosin.

TABLE 1.—Exports of turpentine (gallons) by various countries, 1901-1910.

Country.	1901	1902	1903	1904	1905
France.....	833,918	925,784	1,975,943	1,459,282	3,179,073
Germany ¹	565,174	502,434	612,052	569,644	520,745
Netherlands.....	941,518	1,288,866	988,049	876,921	972,704
Russia.....	1,483,439	1,516,096	1,887,430	2,163,759	2,504,423
Spain.....	² 1,089,669	² 1,089,669	² 1,089,669	² 1,089,669	² 1,089,669
United States.....	20,627,633	18,620,317	15,651,937	16,426,756	15,614,323
Other countries.....	60,000	60,000	72,000	113,000	90,000
Total.....	25,601,351	24,003,351	22,277,080	22,669,031	23,970,937

Country.	1906	1907	1908	1909	1910
France.....	3,367,337	2,538,689	2,397,686	2,400,228	2,851,038
Germany ¹	460,731	349,552	433,235	380,385	429,499
Netherlands.....	1,400,631	165,771	1,851,918	2,068,870	1,812,021
Russia.....	2,456,844	2,705,255	1,773,655	1,833,377	2,473,311
Spain.....	² 1,089,669	907,429	1,131,140	1,150,493	1,169,615
United States.....	16,182,500	17,176,843	19,433,181	16,061,783	14,252,321
Other countries.....	106,000	95,000	226,000	444,000	576,000
Total.....	25,063,712	25,448,539	27,246,815	24,339,136	23,563,805

¹ Not including free ports prior to Mar. 1, 1906.² Average 1907-1910.

TABLE 2.—Imports of turpentine (gallons) by various countries, 1901-1910.

Country.	1901	1902	1903	1904	1905
Austria-Hungary.....	1,762,578	1,821,949	1,739,705	2,071,834	2,021,465
Canada.....	923,370	908,151	890,135	910,443	948,100
Germany.....	8,435,688	8,077,409	8,300,166	8,438,872	8,539,824
Italy.....	658,410	663,187	771,457	816,621	687,284
Netherlands.....	1,895,548	3,245,583	2,729,788	2,220,134	2,248,032
United Kingdom.....	9,701,051	7,942,324	8,012,184	7,907,419	7,693,933
Other countries.....	2,118,199	1,655,645	1,931,217	2,453,412	2,199,739
Total.....	25,594,844	24,314,248	24,374,652	24,818,735	24,338,377

Country.	1906	1907	1908	1909	1910
Austria-Hungary.....	2,218,072	2,291,131	2,409,689	2,439,635	2,502,527
Canada.....	1,011,283	1,028,936	1,081,180	1,141,238	1,044,734
Germany.....	9,966,690	8,986,011	10,088,770	9,764,051	8,659,883
Italy.....	948,162	921,278	1,020,117	824,643	855,538
Netherlands.....	2,711,769	3,035,966	3,932,317	2,785,377	2,696,243
United Kingdom.....	7,673,758	7,515,293	8,656,464	6,522,833	7,041,316
Other countries.....	3,174,227	3,172,429	2,947,196	2,478,389	2,848,791
Total.....	27,703,961	26,951,074	30,135,733	25,956,166	25,649,032

TABLE 3.—Exports of rosin (barrels) by various countries, 1901-1910.

Country.	1901	1902	1903	1904	1905
Austria-Hungary.....	12,933	12,066	11,884	12,955	12,044
Germany.....	150,918	120,558	159,115	162,918	165,066
Netherlands.....	233,833	267,343	225,136	299,794	209,085
Russia.....	140,289	135,978	125,565	147,971	174,273
Spain.....	10,000	17,143	23,929	32,143	56,429
United States.....	2,691,523	2,629,519	2,563,977	2,501,521	2,258,126
Other countries.....	1,168	2,095	5,820	9,765	20,266
Total.....	3,240,664	3,184,702	3,115,426	3,167,067	2,895,829

Country.	1906	1907	1908	1909	1910
Austria-Hungary.....	11,266	10,784	9,399	8,188	7,255
Germany.....	164,602	196,495	217,706	171,497	198,865
Netherlands.....	284,104	273,832	309,885	202,249	199,335
Russia.....	147,161	174,607	189,826	90,410	137,661
Spain.....	51,786	61,967	60,394	53,183	80,602
United States.....	2,481,269	2,636,149	2,601,181	1,984,525	2,269,339
Other countries.....	20,045	22,455	18,340	20,114	43,294
Total.....	3,160,233	3,376,289	3,356,731	2,530,166	2,936,351

TABLE 4.—Imports of rosin (barrels) by various countries, 1901–1910.

Country.	1901	1902	1903	1904	1905
Austria-Hungary.....	284,128	208,749	257,576	231,515	223,149
Canada.....	140,639	28,001	57,033	93,118	67,525
Germany ¹	839,724	705,489	844,585	834,069	743,905
Russia.....	237,030	234,733	241,735	234,261	213,489
United Kingdom.....	692,980	721,487	655,742	712,778	632,181
Other countries.....	777,341	870,925	770,066	935,580	842,835
Total.....	2,971,842	2,769,384	2,826,737	3,041,322	2,723,084

Country.	1906	1907	1908	1909	1910
Austria-Hungary.....	261,980	265,415	294,012	250,822	253,425
Canada.....	68,454	78,058	60,729	82,026	85,438
Germany ¹	840,351	884,393	1,022,197	774,308	857,970
Russia.....	216,361	242,655	269,738	201,176	223,629
United Kingdom.....	624,988	634,051	613,209	530,192	568,914
Other countries.....	859,326	953,244	1,031,975	826,077	901,688
Total.....	2,871,460	3,057,816	3,291,860	2,664,601	2,891,064

¹ Not including free ports prior to Mar. 1, 1906.

Tables 5 and 6 show the growth and present magnitude of the turpentine and rosin industry in the United States and the large amount of capital involved in producing and exporting naval stores. It would seem from Table 6 that the production of turpentine and rosin in this country has reached its maximum, and this conclusion is further borne out by a survey of the stumpage supply still available for naval stores operations (p. 41). Table 7 shows the exports of naval stores by States.

TABLE 5.—Number of establishments and quantity and value of turpentine and rosin produced—United States.

[Figures taken from reports of the Bureau of the Census.]

Year.	Number of establishments.	Turpentine.		Rosin.		Combined value of turpentine and rosin.
		Gallons.	Value.	Barrels.	Value.	
¹ 1913		32,000,000		3,815,000		
¹ 1912		34,000,000		4,000,000		
¹ 1911		31,900,000		3,800,000		
1910		27,750,000	\$17,680,000	3,651,000	\$18,255,000	\$35,935,000
1909	1,585	28,941,000	12,654,000	3,258,000	12,577,000	25,231,000
1908	1,696	36,589,000	14,112,000	4,288,000	17,795,000	31,907,000
1907	1,629	34,181,000	18,283,000	3,999,000	17,317,000	35,600,000
1904	1,287	30,687,000	15,170,000	3,508,000	8,726,000	23,896,000
1900	1,503	38,488,000	14,960,000	2,563,000	5,129,000	20,090,000
1890	670					² 8,077,000
1880	508					² 5,877,000
1870	227					² 3,585,000
1860	625					² 6,468,000
1850	856					² 2,856,000
1810		122,900	176,650	³ 2,000		

¹ According to Naval Stores Review of Apr. 4, 1914.² Combined value of all naval stores.³ Includes pitch.

TABLE 6.—Quantity and value of spirits of turpentine and rosin exported, 1860-1913.

[Figures from the Bureau of the Census.]

Year (ending June 30).	Turpentine		Rosin.	
	Gallons.	Value.	Barrels.	Value.
1913	21,039,597	\$8,794,656	2,806,046	\$17,359,145
1912	19,599,241	10,069,135	2,474,460	16,462,850
1911	14,817,751	10,768,202	2,189,607	14,067,335
1910	15,587,737	8,780,236	2,144,318	9,753,488
1909	17,502,028	7,018,058	2,170,177	8,004,838
1908	19,532,583	10,146,151	2,712,732	11,395,126
1905	15,894,813	8,902,101	2,310,275	7,069,084
1903	16,378,787	8,014,322	2,396,498	4,817,205
1900	18,090,582	8,554,922	2,369,118	3,796,367
1890	11,248,920	4,590,931	¹ 1,601,377	¹ 2,762,373
1880	7,091,200	2,132,154	¹ 1,040,345	¹ 2,368,180
1870	3,246,697	1,357,302	¹ 583,316	¹ 1,776,625
1860	4,072,023	1,916,289	¹ 770,652	¹ 1,818,238

¹ Turpentine included with rosin.

TABLE 7.—Exports of spirits of turpentine and turpentine and rosin by decimal years, 1860-1900.

[Figures from Twelfth Census of the United States, No. 126.]

State.	1900		1890		1880	
	Spirits of turpentine.	Turpen- tine, rosin, and pitch.	Spirits of turpentine.	Turpen- tine, rosin, and pitch.	Spirits of turpentine.	Turpen- tine and rosin.
	Gallons.	Barrels.	Gallons.	Barrels.	Gallons.	Barrels.
Alabama.....	153,018	58,646	210.			22,373
Alaska.....		7				
California.....	45	535		25	6,055	125
Delaware.....						1,375
Florida.....	795,267	243,452	1,742	940	25,728	12,215
Georgia.....	14,623,328	1,408,928	7,251,929	841,217	570,549	91,909
Louisiana.....	212,031	47,890	599	1,128	276	5,089
Maine.....	34,103	831	4,062	79	90	528
Maryland.....	111	174,416	3,002	50,928	754	7,623
Massachusetts.....	2,044	18,359	29,418	7,038	50,915	3,612
Michigan.....	307,716	3,879	5,434	1,939	7,639	103
Minnesota.....		5	7,053	5	362	17
Mississippi.....		2				10
Montana and Idaho.....	11					
New York.....	1,630,164	252,801	894,287	267,801	1,105,100	227,746
North Carolina.....	53,974	139,767	1,751,270	304,100	* 3,630,009	497,456
North and South Dakota.....	39,649	1,774				
Ohio.....	40					
Oregon.....			650			
Pennsylvania.....	121	144	500	1,201	1,443	7,974
Rhode Island.....						
South Carolina.....		21,248	1,293,389	140,399	1,691,447	158,563
Texas.....	659	126	1,515	412	762	42
Vermont.....	235,776	15,631			41	
Virginia.....				2,491	30	3,585
Washington.....	2,525	923	3,860			
Wisconsin.....				1		
Total.....	18,090,582	2,389,364	11,248,920	1,619,704	7,091,200	1,040,345

TABLE 7.—Exports of spirits of turpentine and turpentine and rosin by decimal years, 1860-1900—Continued.

State.	1870		1860	
	Spirits of turpentine.	Turpentine and rosin.	Spirits of turpentine.	Turpentine and rosin.
	Gallons.	Barrels.	Gallons.	Barrels.
Alabama.....	462	885		500
California.....	1,965	76	1,280	2
Connecticut.....			640	10
Florida.....	90	518		
Georgia.....		519	137	134
Louisiana.....	7,558	8,423	11,197	18,909
Maine.....		41		160
Maryland.....	6,104	30,626	38,086	20,268
Massachusetts.....	52,511	11,435	123,163	16,605
Michigan.....		32		
Minnesota.....		1		
New York.....	796,824	464,538	2,816,768	562,253
North Carolina.....	2,042,756	33,212	736,948	77,851
Ohio.....		1		
Pennsylvania.....	544	3,063	25,511	19,845
Rhode Island.....			200	534
South Carolina.....	337,530	25,279	315,099	50,753
Texas.....	273	318		
Vermont.....		2		80
Virginia.....	80	4,347	3,000	2,748
Total.....	3,246,697	583,316	4,072,023	770,652

NOTE.—The exports of turpentine and rosin from a State bear no relation to the amounts actually produced within the State, but to the possession of shipping centers for the naval stores trade.

COMMERCIAL UTILIZATION OF PRODUCTS.

TURPENTINE.

Paints and varnishes.—The greater portion of the turpentine produced finds its way into paints and varnishes. The three main classes of varnishes are spirit varnishes, linseed-oil varnishes, and turpentine varnishes. The turpentine varnishes are made by dissolving resins, such as amber, copal, etc., in hot turpentine and are tough and flexible. Linseed-oil varnishes are often diluted with turpentine.

Turpentine is used in paints and varnishes chiefly as a thinner, of which the properties demanded are solvent action, oxidizing power, penetration, and proper evaporation.

Print goods.—Turpentine finds an important use in the manufacture of cotton and woolen print goods in preventing "bleeding," or running together of colors, where several colors are printed at the same time. It also prevents the color from penetrating the fabric, which is particularly important in the case of woolen goods if unevenness of the material is to be avoided.

Camphor.—Many attempts have been made to produce camphor from turpentine on a commercial scale, but so far none has been entirely successful. However, terpineol, terpin hydrate, and similar bodies are manufactured from turpentine in considerable quantities.

Rubber industry.—Turpentine is important as a solvent for rubber, caoutchouc, and similar substances.

ROSIN.

Rosin is employed extensively in the manufacture of soap, paper, oilcloth, linoleum, sealing wax, fly paper, printing inks, roofing materials, brewer's pitch, electric wiring, lubricating compounds, medicinal preparations, etc.

Rosin soap.—Rosin has the property of combining with alkalies, such as caustic soda and potash, to form "soaps" which are readily soluble in water. Their color is yellowish or yellowish-brown, depending on the color of the rosin used. At ordinary temperatures rosin soaps have the consistency of butter, and on account of their relative cheapness, are usually added to the hard soaps made from tallow. In themselves, however, rosin soaps possess valuable properties, and their addition to tallow or other hard soaps can not always be considered an adulteration.

Resin driers.—The metallic salts of the resin acids are known as driers. They are made by adding a solution of the salt of a metal such as manganese to a solution of "rosin soap" when the insoluble manganese resinate precipitates; or the rosin is fused with the metallic oxide. These metallic resinates, known as "Japan driers," cause the oxidation or "drying" of oil paints and varnishes and are extensively used for this purpose. The lead and manganese resinates are used most frequently.

The various enamels used in ceramics consist of resinates of the various heavy metals. The resinates are dissolved in turpentine and the resulting solution painted on the earthenware, after which the vessels are "fired."

Rosin size.—One of the most important uses of rosin is as a "size" or coating for writing or printing papers, which must take ink. A rosin soap, containing about 3 pounds of rosin to 1 of soda, is added to the pulp in the hollander, and after that a solution of alum. The latter decomposes the rosin soap, and the result is a precipitate of free rosin and some alumina which becomes entangled in the fibers of the paper. When the paper is passed over hot calendar rolls in finishing, the rosin fuses to a smooth, varnish-like layer on the surface.

Brewers' pitch.—Barrels intended to hold beer or other fermented beverages are coated with brewers' pitch, which renders the barrels easy to clean and improves the taste of the beer. The pitch is made of pure rosin, with the addition of a certain amount of turpentine or refined rosin oil to prevent brittleness. Some manufacturers make the pitch supple by adding rosin soap.

Products of the distillation of rosin.—When rosin is heated at temperatures above its melting point it decomposes¹ into gases and oils.

The lightest of these oils, rosin spirit or pinoline, is used as an illuminant, and also as a solvent, especially as a substitute for turpentine. The heavier rosin oils are used in the manufacture of printing inks and lubricants. Wagon grease is often made by boiling rosin oil with lime.

The distillation of rosin is carried out on a large scale in Germany and France, the darker grades of rosin imported from America being used for this purpose. Comparatively little is distilled in the United States.

Lampblack.—Rosin oil can be used for the manufacture of lampblack which, from the standpoint of color and minuteness of division, is of the highest quality. The finest grades are used in the manufacture of india ink, in lithography, and in artistic printing with copper plates.

FORMATION AND FLOW OF RESIN IN THE LIVING TREE.

Resin suitable for the production of naval stores is found only in in coniferous trees. Moreover, only pines yield resin abundantly, and of these only two species, longleaf pine (*Pinus palustris*) and, to a small extent, slash pine² (*Pinus heterophylla*) are tapped in the United States.

No universally accepted theory dealing with the formation of resin has as yet been advanced. It is generally conceded, however, that resin is formed as a by-product during the transformation of food materials, such as starch, into woody tissue. The resin is stored in two systems of elongated passages or resin ducts. In one system the ducts are parallel to the pith of the tree; in the other they lie horizontally in radial planes. The ducts form in the growing tissue or cambium layer just beneath the bark, the two systems intersecting to form a continuous network of resin passages.

When the cambium layer is cut the growth of tissue near the wound is stimulated, and the number of resin ducts, and consequently the amount of resin formed, is considerably increased. The area in which additional or secondary resin ducts are formed apparently extends from 2 to 3 inches above and to a lesser distance below the wound.

¹ The products resulting from distillation and their percentages are as follows:

Losses (rosin adhering to walls of still) per cent..	1.0	Light oil (turbulent).....per cent..	5.0
Gases evolved.....do....	9.0	Light oil, hearts.....do....	58.0
Acid water.....do....	3.5	Blue oil and red oil.....do....	16.0
Rosin spirit or pinoline.....do....	3.5	Residue, coke.....do....	4.0

² Slash pine is of comparatively infrequent occurrence, but is tapped wherever found on areas being turpintined.

The additional ducts require from 2 to 4 weeks for their formation full of resin.

If a new cut is made just above the old one, after the additional ducts have had a chance to form, the flow will show a large increase over that obtained from the original wound, due to the additional ducts.

Depth of cut.—Since the additional ducts form only in the cambium layer, and since they yield by far the greater part of the resin, cutting deeper than this layer induces but little additional flow. In commercial operations the depth of the cuts or "streaks" runs from one-half to one inch. Such streaks are, of course, much deeper than necessary, and to just that extent tend to reduce the vitality of the tree and, in consequence, its ability to give a sustained flow. Tests have shown that a greater average flow over a four-year period can be obtained from trees with streaks 0.4 inch deep than from trees with streaks 0.7 inch deep. In any case, shallow streaks give fully as large a flow of resin as deep ones, when the period of tapping extends over two years or more. The tools used at present, however, make it difficult to cut shallow streaks, while the custom of deep chipping is pretty firmly established through long usage.

Height of chip.—When a new "streak" is made the flow of gum is at first comparatively rapid, but gradually decreases until at the end of a week it has practically ceased. The diminution of flow is presumably caused by the gradual hardening of the resin in the exposed ends of the ducts, which results in plugging them. It then becomes necessary to chip again. In deciding how thick a chip should be taken off, or how much the "face" or scar should be increased in height to give a new flow, it should be remembered that the bulk of the resin is produced in the region between the wound and a point about two inches above it. For this reason, no more of the wood immediately above the old wound should be removed than is necessary to open up the ends of the resin ducts in which the gum has hardened. Since in the space of a week the resin does not harden in the ducts for a distance greater than one-fourth inch from the surface of the cut, a chip that increases the height of the face one-fourth inch is all that is necessary. In practice, the vertical height of the new streak frequently exceeds 1 inch, thus eliminating practically one-half of the wood where most of the resin is being produced, and decreasing the productive period of the tree four times as rapidly as necessary. With the present type of tool it is difficult to cut a one-fourth inch streak, and, moreover, the difficulty of changing an old established custom again presents itself. The chipping tool should always be sharp. A dull edge tends to crush the ends of the resin ducts instead of cutting them clean, thus preventing a free flow.

Frequency of chipping.—Table 8 shows the rate with which the gum flows.

TABLE 8.—Rate of exudation of gum from "chipped" longleaf pine.

Day.	Grams of gum.	Total exudation.	Day.	Grams of gum.	Total exudation.
		<i>Per cent.</i>			<i>Per cent.</i>
First.....	113.0	67.26	Fifth and sixth.....	9.0	5.36
Second.....	22.5	13.39	Seventh.....	1.0	0.59
Third.....	13.5	8.04			
Fourth.....	9.0	5.36	Total.....	² 168.0	100.00
Fifth.....	(¹)	-----			

¹ No weighing.

² 168 grams equal 0.37 pound.

It is seen that 88 per cent of the total flow occurs during the first three days. As the resin ducts become plugged with coagulated or crystallized gum the flow gradually ceases, and the gum thereafter produced is stored in the resin ducts until the ends are again opened. When the ducts immediately above the wound become full, the resin tends to diffuse or soak into the wood further removed from the bark. This diffused resin does not drain out when the tree is wounded, and for this reason chipping should be done often enough to insure that the active ducts immediately beneath the bark and above the wound will not remain full of gum. On the other hand time should be allowed between chippings for a new supply of gum to form. In practice, trees are chipped once a week. It is possible that more frequent chipping would give a greater yield of gum for a short period (one or two years), but at the same time it might further reduce the vitality of the tree and so result in a smaller total yield over a longer period. The increased yield, moreover, must be enough to justify the additional expense. Experiments are needed to show how the rate of flow is affected by frequency of chipping in operations extending over different periods of years.

Size and number of faces.—The scar on the tree caused by successive chippings is usually about 14 inches wide, and is known as the "face." Wounding the tree, of course, diminishes its vitality by interfering with the transmission of water from the roots to the leaves and of nutritive matter from the leaves to the roots. When a small tree, 8 or 10 inches in diameter, is chipped, it usually either dies outright or its further growth is greatly retarded, even though the width of the face is kept at the minimum.

PRINCIPLES UNDERLYING THE DISTILLATION OF CRUDE GUM.

The crude gum was formerly distilled without the addition of water; in consequence the quality of the resulting turpentine and rosin was poor. The yield of turpentine was very low, but it was impossible to increase it without coloring the liquid yellow with the

decomposition products of the rosin. Other conditions being the same, the question of obtaining water-white turpentine and rosin depends largely on the temperature.¹ The introduction of the practice of using water during the distillation increased the yield and quality of the turpentine and resulted in rosin of a lighter color.

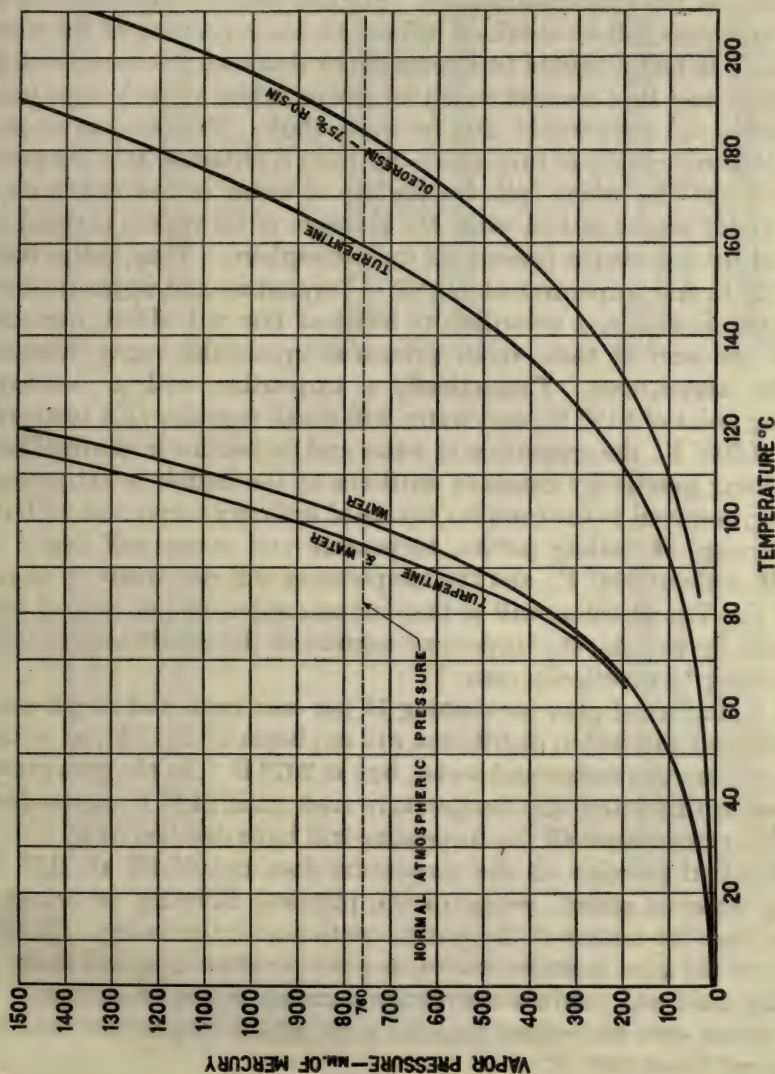


Fig. 2.—Relation of vapor pressure and temperature for turpentine, water, and mixtures.

Turpentine begins to boil at about 313° F.² and the greater portion of fresh turpentine distills between 317° F. and 324° F. (See fig. 2). If an attempt is made to distill turpentine direct from a gum contain-

¹ It should be borne in mind, however, that it is impossible to make light rosin from scrape and dip from old boxes or when the gum contains large amounts of trash by following the ordinary methods of production. See p. 27.

² 203° F. = 95° C. 302° F. = 150° C. 324° F. = 162° C. 207° F. = 97° C. 313° F. = 156° C. 363° F. = 184° C. 212° F. = 100° C. 317° F. = 158° C. 392° F. = 200° C.

ing 75 per cent rosin and 25 per cent turpentine, the turpentine in the absence of water will not begin to boil at 313°F. , but at about 363°F. , owing to the presence of the rosin. Rosin does not begin to decompose perceptibly until a temperature of about 392°F. is reached, but after the turpentine in the gum begins to distill off at 363°F. , the temperature of the gum rises rapidly. For this reason, only a portion of the turpentine will be obtained before the decomposition of the rosin begins. In fact, it would be impossible to obtain all the turpentine in the gum, and that secured would be yellow, while at such high temperatures the rosin would also be quite dark. Practice has shown that the best quality of turpentine and rosin is obtained at a temperature of 302°F. , which calls for the use of water in the distillation.

A liquid begins to boil when the pressure of its vapors is equal to or slightly exceeds the pressure of the atmosphere. Thus, water boils at 212°F. and turpentine at 313°F. Turpentine and water are non-miscible liquids, and according to physical law will distill together when the sum of their vapor pressures equals the vapor pressure of the atmosphere. Theoretically a turpentine, with a constant boiling point of 313°F. , and water will distill together at a temperature of 203°F. , the proportion of water and turpentine in the distillate remaining practically constant until one of the liquids is exhausted. Owing, however, to the complex nature of ordinary turpentine with its wide range of boiling points, turpentine and water will begin to distill at about 203°F. , and the temperature will rise finally to about 212°F. The distillate will at first contain about 60 per cent of turpentine by volume, the turpentine content of the distillate gradually decreasing to practically zero.

In a mixture of gum (containing 75 per cent rosin and 25 per cent turpentine) and water, distillation will not begin at 203°F. , as in the case of pure turpentine and water, but at 207°F. As the gum grows poorer in turpentine the temperature rises until 212°F. is reached. At this temperature all the turpentine will have distilled over.

In actual practice all the turpentine does not distill at 212°F. when water is added, owing to the physical difficulty of bringing more than the surface of the gum in contact with the water. On this account the gum must be heated to a temperature that will make it readily fluid and produce convection currents in lieu of stirring. If live steam were introduced into the mass all the turpentine could be removed below 212°F.

COMMERCIAL METHODS OF COLLECTING CRUDE GUM.

BOX SYSTEM.

Cutting the boxes.—The first operation in turpentinizing by the box method consists in cutting a cavity (fig. 3) into the base of the tree for holding the crude gum. This cavity, called the "box" is cut

during the winter months. The work is performed by a squad of six or seven negroes under an experienced overseer, who tallies the boxes. The cutting is done by piecework, and each negro has a number which he calls as soon as he has finished a box. The tool used is an ax with a long narrow blade. An experienced man will cut a box

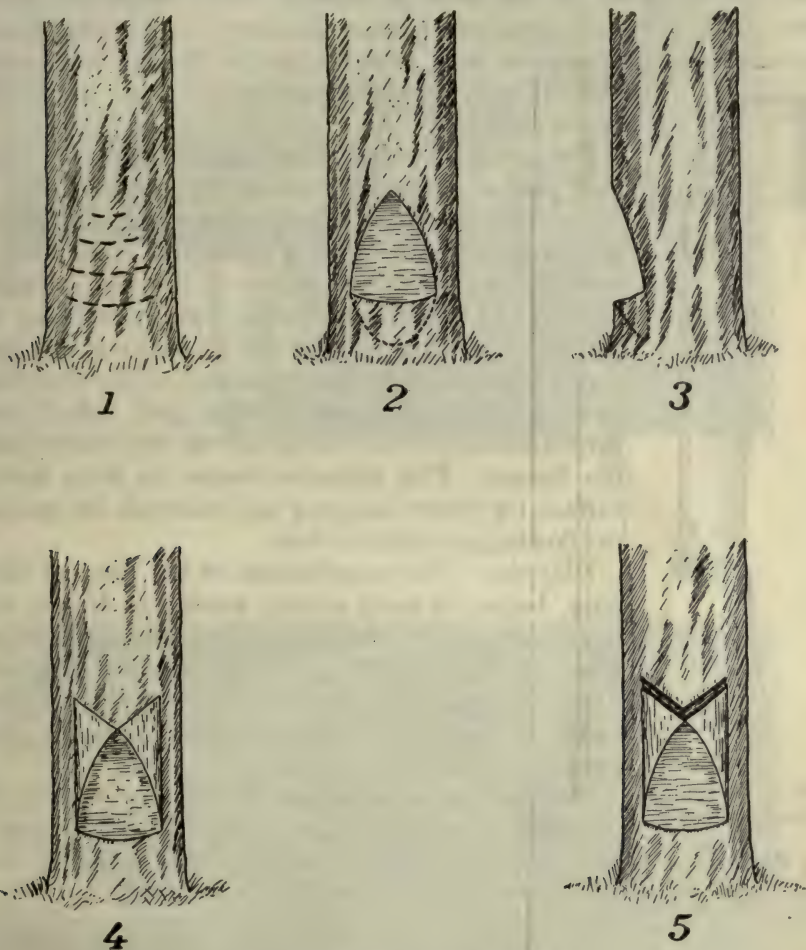


FIG. 3.—Operations in cutting a "box."

1. First step in cutting box. Gashing tree.
2. Finished box. Front view.
3. Finished box. Side view.
4. Cornered box.
5. First streak.

with surprising neatness in from four to eight minutes. The dimensions of the box vary somewhat with the size of the tree. Usually a box is 12 to 14 inches wide, 7 inches deep, $3\frac{1}{2}$ to 4 inches from front to back, and holds about 3 pints. Shaped like a distended pocket, it is cut into the base of the tree 8 to 12 inches above the ground,

although in second-growth timber this distance may be only from 5 to 6 inches.

The position of the box depends on the configuration of the tree. If the latter leans, as is usually the case, the first box is placed on the side opposite to the direction in which it leans, which is generally the position occupied by the most prominent root. When additional boxes are cut on a leaning tree, the loss occasioned by the gum falling outside the box increases each year the tree is bled; in some cases little, if any, resin reaches the box from the fourth year's chipping.

Cornering.—Two or three weeks before the chipping season opens the "boxes are cornered" (fig. 3 (4)). The operation, which is done with an ordinary ax, consists in removing a triangular chip about 1 inch thick above the corners of the box, a right-handed and a left-handed man usually working together. The chip is removed by making one gash which rises obliquely from the apex of the triangular opening of the box and another gash which rises perpendicularly from the corner of the box to meet the former. This operation serves to form both a surface for future chipping and channels for guiding the flowing gum into the box.

Chipping.—The scarification of the tree, or chipping, begins in early spring, usually in March, and continues each week up to October or November, when the flow of gum practically ceases. The number of chippings is usually 32 per season, although owing to weather or labor conditions it may vary between 28 and 35.

FIG. 4.—Hack with handle and weight.

The instrument used for chipping or making the "streak" is called a "hack" (fig. 4). It consists of a flat, steel blade $2\frac{1}{2}$ inches wide, bent into the shape of a U, measuring an inch between the sides.¹ The blade is fastened at right angles to one end of a wooden handle 18 inches long and 2 inches in diameter, to the opposite end of which is attached a pear-shaped iron weight weighing from 5 to 7 pounds; the blade and handle weigh about 1 pound.

The chipper (Pl. I, fig. 1) stands directly in front of the face and removes with the hack two strips of wood and bark one-half to three-fourths of an inch wide and one-half to $1\frac{1}{2}$ inches deep, parallel with the oblique gashes made in cornering. The removal of the two strips constitutes the "streak," which is in the shape of a V, having an angle of about 95° . The apex of the angle is called the "peak" and

¹ Three sizes of hacks are made. The blade described is for a No. 1 size hack.



FIG. 2.—CUP AND GUTTERS.

Blaze below first streak 8 by 16 inches. Distance from gutter to peak of first streak $8\frac{1}{2}$ inches, of which 7 inches, equivalent to 14 streaks, represents a loss of possible chipping height.



FIG. 1.—CHIPPING.



"PULLING."

This tree originally boxed; cups substituted later.

lies directly above the center of the box. The hack is given a quick, swinging motion, and the momentum furnished by the iron ball enables one side of a streak to be made with only two or three blows. Chippers are paid from 75 cents to \$1 per thousand streaks, according to the condition of the crop and the scarcity of labor.

After the first two seasons the increased height of the face makes the use of a hack impracticable, and a "puller" is used in its place. This tool resembles the hack, except that the blade is closed and provided with a long handle. The streak is made by a steady pull (Pl. II) rather than with a quick hacking motion. After the fourth season the face is usually abandoned, since at that time a height of 8 feet is attained, beyond which it is not profitable to work a tree. Old faces 12 feet in height have been noted in Georgia, and others even higher in North Carolina and South Carolina.

Dipping.—The boxes fill with gum in three or four weeks and are "dipped" or emptied about seven times a season. The workman uses a tool called a "dipper", which has a flat trowel-shaped blade about $7\frac{1}{2}$ inches long and $5\frac{1}{2}$ inches wide. This dipper is thrust into the box under the gum, which is removed by a quick upward and outward motion and flipped into a portable bucket. Considerable skill is required to prevent loss of gum during the transfer. The bucket is emptied into barrels placed at convenient points throughout the woods. These barrels, provided with removable heads, are closed after filling, rolled upon wagons by means of skids, and taken to the still. Dippers receive from 50 cents to \$1 per barrel, according to the nature of the territory covered.

Scraping.—A certain amount of the gum does not reach the box, partial evaporation of volatile oil leaving it too viscous to flow. Gum which is perfectly homogeneous and transparent immediately after exudation soon becomes opaque from the separation of white crystals of the resin acids, and doubtless the greater portion of the "scrape" results from the adherence of these separated crystals to the face. The amount of this hardened gum naturally increases with the height of the face. "Scrape" is essentially a product of longleaf pine (*Pinus palustris*), since slash pine (*Pinus heterophylla*) forms but small amounts, which it does not pay to collect. The scrape contains about half as much turpentine as the "dip" and gives a darker resin under similar conditions.

The scrape is collected but once a year—at the end of the season. The tools used, called "scrapers," are of two types. One type, the "pusher," has a flat, rectangular blade 4 inches long by $4\frac{1}{2}$ to 5 inches wide. This is used during the first two years, the scrape being removed by downward thrusts of the tool. In most cases the necessary violence of the thrust results in removing large chips of wood along with the scrape. Another type of scraper has a blade shaped

like an equilateral triangle and in use is given a pulling motion downward.

The scrape is usually collected and distilled before the last dipping is made, since at the end of the season the danger of fire breaking out in the woods and burning the faces is very great. The dip in the boxes is not so great a source of danger, since it is usually more or less covered with water. In course of removal the scrape is considerably disintegrated, and if stored in the rosin barrels for any length of time the turpentine evaporates rapidly.

A wooden box, called a "scrape box," is used to receive the detached scrape. This box is about $2\frac{1}{2}$ feet square and open at the top and at one end. The bottom at the open end is rounded inward and provided with an apron of burlap to form a close contact with the tree and prevent loss of scrape. The legs project sufficiently above the sides to serve as handles for dragging the box from tree to tree, though sometimes the box is provided with wheels. A box will hold from 100 to 150 pounds of scrape, which is transferred to rosin barrels and hauled to the still.

Raking.—After the crude gum has been collected the trash surrounding the base of the tree is "raked" away for a distance of $2\frac{1}{2}$ to 3 feet to guard against fire. This operation is performed late in the fall, the tool used being a hoe with a broad, heavy blade. The turpentine woods are intentionally burned over once each season, to afford better forage for stock the following spring, to reduce the risk from accidental fires, and to remove brush and other materials which impede the workmen.

Crops.—The tracts of timber to be turpented are divided into sections called "crops," a full crop consisting of 10,500 boxes.¹ Since each tree receives from one to four boxes, 4,000 to 5,000 trees, covering an area of 200 to 250 acres, are required to make one crop. For convenience in working, the crop is further divided into drifts, whose boundaries are defined by lines blazed on the trees. Each drift contains about 2,100 boxes, although this number varies considerably.

The average still has a capacity of 15 to 20 barrels, so in order to make two distillations per day with a still holding 20 barrels of crude gum the operator must work 20 crops, covering an area of four to five thousand acres. It is seldom profitable to work less than five crops.

CUP SYSTEMS.

Historical.—Until recent years the box system was the only one used in the United States for collecting resin. While no recent figures are available, it is probable that at present the number of cups

¹ In practice a "crop" consists of the number of faces a man can chip in from four to five days. Consequently a "crop" may vary from 7,000 to 10,000, owing to the topography of the country or density of the stand.

in use exceeds the number of boxes. During the past two or three years, however, it is estimated that cups have been hung on 75 per cent of the trees tapped.

The need of replacing the box with a cup hung against the tree was felt many years ago.

Mr. A. Pudigon patented a substitute for the box in 1868, and the device received a commercial test at Monks Corner, S. C., but was soon abandoned for some unknown reason. From that time on numerous substitutes have been invented, but none patented prior to 1903 proved a commercial success.

The first systematic attempt to improve the method of collecting gum was made at Bladenboro, N. C., by W. W. Ashe in 1894. A comparison on a limited scale was made between the French cup and gutter system and the box system, and the results showed a gain for the former of over 20 per cent in the value of the products collected.

The preliminary experiments begun in 1901 by Dr. Charles H. Herty, of the Forest Service, and continued in 1902, mark the turning point in the method of collecting crude gum. Cups were first used on an extensive scale in 1904, and since that time their use has become more or less general.

Classes of cup systems in use at present.—The cup systems may be divided into four classes.

Class 1. (Plates III and IV.) The gum flowing down the face is guided into the cup by means of two galvanized-iron gutters inserted in cuts in the tree. These gutters are 2 inches wide and from 6 to 12 inches long, depending on the size of the tree, and are bent into an obtuse angle. Sufficient bark and sapwood are removed from the tree to form a central vertical ridge with two flat faces on either side of it. The gutters are inserted in inclined gashes made by a broadax in the flat surfaces. It is necessary that these surfaces be flat, in order that the straight edge of the gutter may enter the face along its entire length, so that gum can not flow between the gutter and the tree. The lower gutter is placed so as to project at least two inches beyond the other at the center of the ridge, in order to guide the gum into the cup, which is hung just below the lower gutter on a nail. The cups are of galvanized iron or of clay, and vary in shape. Those resembling an ordinary flowerpot are the most common. Their capacity is 1, $1\frac{1}{2}$, or 2 quarts. The blazes made for inserting the gutters extend below the latter and produce a flow of resin which is not only wasted but serves to coat the base of the tree, and thus makes the face more susceptible to fire. The workman frequently makes the blaze too large, as is shown in Plate III, figure 1, and there is a tendency in placing the gutters to spread them too far apart, losing in many cases as much as 20 inches of chipping surface. It is entirely possible to place the cups and gutters on a normal tree so that the first streak

will be 12 inches from the ground (Plate III, fig. 2). In Plate IV, figure 1, the first streak is 17 inches higher than necessary. On the basis of one-half inch streaks this means that a height sufficient for 34 chippings, or for a whole season, has been lost.

Class 2. (Plates V and VI.) The gum flowing from the face is guided into the cup by means of a flat oblong piece of galvanized iron, with the ends slightly upturned, called an "apron." The edge of the apron to be inserted in the tree is concave to conform to the tree's shape. In some cases the aprons are made in two pieces, riveted together at the ends, so as to allow them to be adjusted to the curvature of each particular tree. They are also made in two separate pieces. The aprons are inserted in a horizontal gash at the base of the tree made with a broadax having a flat blade with a concave edge. When inserting the apron a small blaze about 6 inches wide and 2 inches high is generally made to remove objectionable bark. The broadax is held horizontally against the blaze, with the head slightly downward, by one man, while another drives it into the tree with a maul. The ax is then withdrawn and the apron inserted.

A recently introduced apron is lunar in shape, the concave edge being provided with stiff teeth. This apron can be driven directly into the tree, obviating the necessity of blazes or gashes.

The cups are made of galvanized iron or clay and hold about a quart. Their general shape is that of an oblong box 12 inches by 3 inches at the top, and about 3 inches deep. They are slightly larger at the top than at the bottom, and are sometimes shaped to conform to some extent to the curvature of the tree. The cups are sometimes hung from the apron by means of small hooks which engage an extension on either end of the apron, or they may be supported on a nail driven into the tree beneath the apron. The likelihood of the fasteners becoming clogged by gum is obviated by the use of nails as supports. In hanging this class of cups large blazes are not necessary, and if properly hung practically all the gum flowing from the tree reaches the cups. As the aprons occupy but little vertical space and the cups are comparatively shallow, a distance of 12 inches from the ground to the first streak is ample on normal trees (Plate V, fig. 1). In the case of small trees 10 inches or less in diameter, the use of the 2-piece or riveted apron allows a shallower cut to be made in hanging the cup, as the 1-piece aprons have such a large curvature that they require a deep cut in small trees (Plate VI, fig. 1) to prevent escape of gum at the sides. On large timber, of course, this difficulty does not occur.

Class 3. (Plate VII, fig. 1.) The cup is so constructed as to obviate the necessity of using a gutter or apron. In order to hang it, several



FIG. 2.—PROPERLY HUNG CUP AND GUTTERS.

Twelve inches from ground to first streak.



FIG. 1.—CUP AND GUTTERS.

Twenty-one inches from ground to first streak, 5 inches between gutters, 6 inches from first streak to lower end of upper gutter. Note large useless blaze 16 by 9 inches below first streak. Nine inches of chipping height have been lost.



FIG. 2.—CUP AND GUTTERS.

Lower gutter projects too far. Five inches from lower gutter to first streak.



FIG. 1.—BADLY HUNG GUTTERS.

Twenty-nine inches from ground to first streak. Seventeen inches, or nearly a year's chipping height, has been lost.

streaks are made at the base of the tree, the last one being a "shade" streak or one which is undercut. The prolonged back of the cup is notched and bent to conform to the chipped surface. The cup is loosely hung on a nail, so as to be readily detachable for dipping. The first chipping is so made as to leave a band of bark and wood $1\frac{1}{2}$ to 2 inches wide over which the resin flows and drips into the cup. The last chipping of the season should be a shade streak, so that at the beginning of the succeeding season the cup may be raised and fitted under it and a strip of bark and wood left as before.

Great care must be used in hanging these cups or the loss of resin will be excessive. It is difficult to make a shade streak close to the ground, and if a square streak is made the resin will flow between the tree and the cup. The cup in Plate VIII, figure 1, was originally hung so poorly that rehangng was necessary in the middle of the first season. The equipment for hanging cups of this class is simpler than in the case of those requiring gutters and aprons, and the necessity of gashing the tree with a broadax is obviated.

Class 4. (Plate VII, fig. 2.) Two gutters of galvanized iron are inserted in a streak just above the cup which is hung on a nail. The gutters are semicircular in section and the ends are so riveted together that the gutter may be adjusted to any angle. To hang the gutter, a shade streak is made and the gutter fitted into it and held in position by means of two nails driven through holes made for the purpose.

This gutter can be hung by one man and no special tools are required. No wounding is necessary, except a single streak; and, owing to the rivet, the gutter will serve readily for small and large timber. Unless care is used in placing these gutters, however, the gum will flow between them and the tree.

Class 5. The gum flows from two holes bored diagonally upward into the tree from a common point. The top of the receiving vessel consists of two caps at right angles, connected by a triangular tube. One cap is placed over the holes bored into the tree, while a glass cup screws into the horizontal cap. When full, the cups are unscrewed and emptied. When the flow ceases, the old holes are reamed out or new ones bored. Experiment has shown that the holes in the tree, as well as the tube in the receiving vessel, soon become plugged with gum, rendering the maintenance of a continuous flow an expensive operation. This method of tapping is worthy of mention, however, since by its means two highly desirable results in naval stores operations are obtained, namely, a pure gum and minimum damage to the timber.

Material and shape of cups.—The great majority of cups on the market are made of clay or galvanized iron. The clay ones are the cheaper, and it is claimed for them that they yield a higher quality

of gum, since clay does not become heated like metal when exposed to the sun and thus cause evaporation of the turpentine. Clay cups are three or four times as heavy as metal cups, however, and much more bulky. They are also more likely to break, both in transit and in handling. On the other hand, they do not rust like the galvanized-iron cups. Rusting not only results in loss of cups, but may also darken the gum.

In shape the cups may be like a flowerpot (Plate I, fig. 2), an oblong box (Plate V, fig. 1), or a flattened cone (Plate VII, fig. 2). With the deep cups, it is claimed, there is less evaporation of gum, on account of the smaller surface exposed. On the other hand, deep cups take up more vertical height on the tree, and are generally considered more difficult to dip. The cone-shaped cups are similar in shape to the interior of the ordinary "box," being so made for the sake of economy, since only one seam is necessary.

Within certain limitations the kind of cup used with a particular gutter or apron is immaterial. Between the cups and gutters now on the market, the greatest room for improvement exists in the case of the latter, though improved aprons and gutters are constantly put on the market.

RELATIVE YIELDS SECURED FROM CUPS AND BOXES.

Experiments made by the Forest Service in Georgia during 1902 showed conclusively that more and better turpentine and rosin can be obtained by the use of cups than by the use of boxes.

The timber studied consisted of a first, second, third, and fourth year crop, one-half of each crop being turpented by the cup system and the other half by the box system. The comparative results are shown in Tables 9 and 10.

TABLE 9.—*Spirits of turpentine from eight half crops, season of 1902, Georgia.*

Half crops.	From dip.	From scrape.	Total.	Excess from cupped trees.	
	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Per cent.</i>
First year:					
Cups.....	1,385.3	205	1,590.3	301.9	23.43
Boxes.....	1,134.7	153.7	1,288.4		
Second year:					
Cups.....	1,087.2	188.2	1,275.4	66.6	5.51
Boxes.....	941.8	267	1,208.8		
Third year:					
Cups.....	726.5	113	839.5	310.1	58.58
Boxes.....	381.9	147.5	529.4		
Fourth year:					
Cups.....	687.2	101	788.2	314.2	66.29
Boxes.....	349.5	124.5	474		

TABLE 10.—*Net rosin sales from eight half crops, season of 1902, Georgia.*

Half crops.	From dip.	From scrape.	Total.	Excess sales from cupped trees.	
First year:				<i>Net.</i>	<i>Per cent.</i>
Cups.....	\$401.72	\$47.72	\$449.44	\$85.51	23.50
Boxes.....	328.40	35.53	363.93		
Second year:					
Cups.....	266.34	49.25	315.59	144.13	84.64
Boxes.....	104.51	66.95	171.46		
Third year:					
Cups.....	171.27	27.44	198.71	132.65	200.80
Boxes.....	39.49	26.57	66.06		
Fourth year:					
Cups.....	167.33	29.23	196.56	132.56	207.13
Boxes.....	36.09	27.91	64.00		

The first year crop mentioned in Tables 9 and 10 was worked for two years longer. The combined yields obtained for the "cupped half" and "boxed half" during the 3-year period of operation are given in Tables 11 and 12.

TABLE 11.—*Spirits of turpentine from half crops, seasons 1902-1904, Georgia.*

Year.	Cups.			Boxes.			Excess from cupped half crop.	Net price per gallon at time of operation.	Value of excess from cupped half crop.
	Dip.	Scrape.	Total.	Dip.	Scrape.	Total.			
	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Cents.</i>	
First.....	1,385.3	205.0	1,590.3	1,134.7	153.7	1,288.4	301.9	40	\$120.76
Second.....	1,103.5	165.0	1,268.5	705.2	226.6	931.8	336.7	45	151.52
Third.....	781.3	136.0	917.3	536.1	190.5	726.6	190.7	45	85.82
Total....	3,270.1	506.0	3,776.1	2,376.0	570.8	2,946.8	829.3		358.10

TABLE 12.—*Net sales of rosin from half crops, seasons 1902-1904, Georgia.*

Year.	Cups.			Boxes.			Value of excess from cupped half crop.
	Dip.	Scrape.	Total.	Dip.	Scrape.	Total.	
First.....	\$401.72	\$47.72	\$449.44	\$328.40	\$35.53	\$363.93	\$85.51
Second.....	286.88	58.24	345.12	132.42	84.08	216.50	128.62
Third.....	212.60	61.65	274.25	124.76	79.70	204.46	69.79
Total.....	901.20	167.61	1,068.81	585.58	199.31	784.89	283.92

RELATIVE AMOUNTS OF SCRAPE FORMED BY THE BOX AND CUP SYSTEMS.

The resin obtained from trees turpented by the box method must flow an increasingly greater distance each year the tree is tapped. As a result the amount of scrape formed is proportionately increased. The proportion of scrape formed by the two systems is shown in Table 13.

TABLE 13.—*Comparison of the amount of scrape formed by the cup and box systems, season 1902, Georgia.*

Half crop.	Net weight of scrape.	Net weight of dip.	Total weight of "gum" dip and scrape.	Percentage of scrape.
	<i>Pounds.</i>	<i>Pounds.</i>	<i>Pounds.</i>	<i>Per cent.</i>
First year:				
Boxes.....	10,315	42,787	53,102	19.4
Cups.....	13,155	51,081	64,237	20.5
Second year:				
Boxes.....	17,120	35,700	52,820	32.4
Cups.....	12,210	42,630	54,840	22.3
Third year:				
Boxes.....	8,580	15,435	24,015	35.7
Cups.....	7,200	28,245	35,445	20.3
Fourth year:				
Boxes.....	7,970	14,385	22,355	35.6
Cups.....	6,635	25,305	31,940	20.8

Scrape is troublesome to collect, yields a low grade of resin, and gives but 11 per cent of turpentine on distillation, while gum collected by the cup system yields about 19 per cent of turpentine.

RELATIVE YIELDS FROM DIFFERENT DEPTHS AND HEIGHTS OF CHIPPING.

In the years 1905 to 1908 the Forest Service carried out experiments to determine the effect of the depth and height of chipping on the yield of resin. Four crops¹ were used in the experiment, designated A, B, C, and D, respectively.

Crop A, taken as the standard, was chipped in the ordinary way, the average depth of chipping being seven-tenths of an inch and the average height five-tenths of an inch.

Crop B was used to test the effect of shallow chipping, the average depth being four-tenths of an inch.

Crop C served to show the effect of narrow chipping, the average height being four-tenths of an inch.²

Crop D was turpentine with reference to the possibility of working the turpentine a second time. The present method consists in exhausting the tree within four years. This crop was chipped in the same manner as crop A, but the minimum diameter of the trees turpentine was limited to 10 inches, as compared to a minimum diameter of 6 inches in crop A; in addition the minimum diameter of the tree to bear two faces was raised from 13 inches in A to 16 inches in crop D; no tree in crop D had more than two working faces.

Table 14 shows the yields from the four crops A, B, C, and D.

¹ Crops of 8,000 faces each were used.

² It was intended to have the height of chip in "C" half that in "A," but in spite of close supervision the chippers cut wider than was desired.



FIG. 1.—CUP AND APRON.

Twelve inches from ground to first streak, $1\frac{1}{2}$ inches from gutter to first streak, 10 inches from ground to top of cup.



FIG. 2.—CLAY CUP AND APRON.

Fourteen inches from ground to first streak, 3 inches from gutter to first streak.

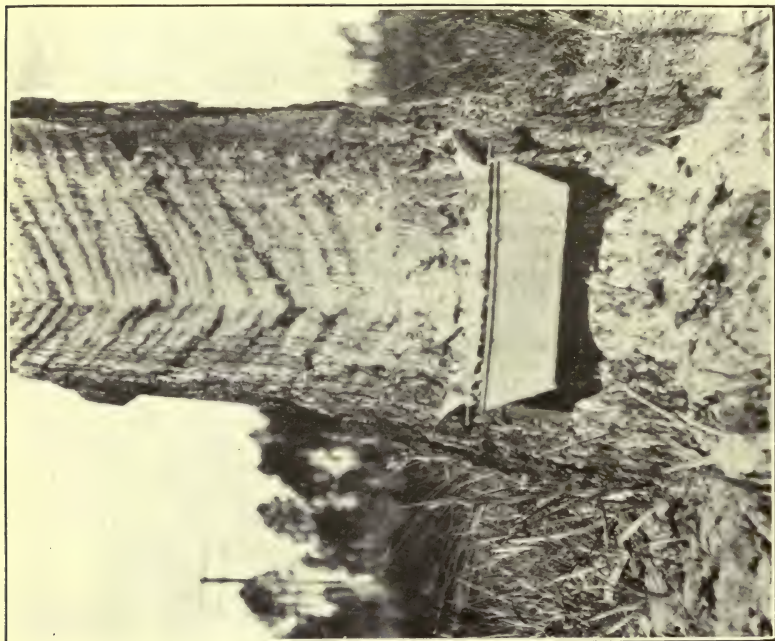


FIG. 2.—CUP AND APRON.

Working second year face without raising cup, thus reducing the efficiency of the cup system.

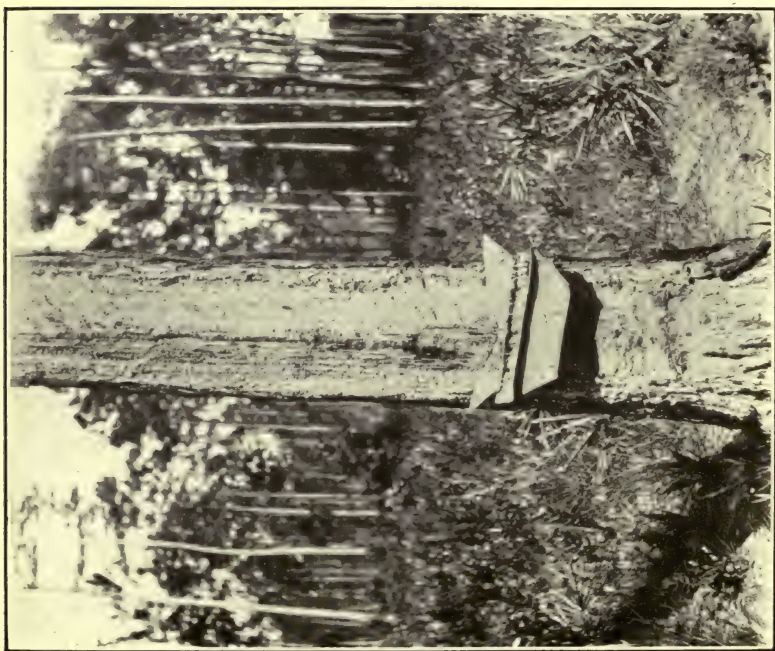


FIG. 1.—CUP AND APRON.

Working face the fourth season and the cup has been raised but once.

TABLE 14.—*Summary of total yields for four years based on the dip and scrape being corrected to the same number of chippings per crop (8,000 faces).*

Crop.	Dip.		Scrape.		
	Yield.	Increase.	Yield.	Increase.	Decrease.
	<i>Pounds.</i>	<i>Per cent.</i>	<i>Pounds.</i>	<i>Per cent.</i>	<i>Per cent.</i>
A.....	206,235		47,742		
B.....	211,911	2.75	44,838		6.08
C.....	214,503	4.01	39,775		16.69
D.....	279,260	35.41	53,915	12.93	

Two crops, G and H, were worked for one year, combining the principles observed under crops B and C, namely, shallow and narrow chipping. Crop X was chipped in the ordinary way. Table 15 gives the yields.

TABLE 15.—*Summary of yields for one year. Crops X, G, and H.*

Crop.	Number of cups.	Number of chippings.	Yield of dip.	Increase.
			<i>Pounds.</i>	<i>Per cent.</i>
X.....	9,880	35	90,094	
G.....	9,880	35	124,292	38
H.....	9,880	35	121,474	35

As seen from Table 15 there is a decided increase in yield by the use of shallow and narrow chipping.

EFFECT OF TURPENTINE OPERATIONS ON TIMBER.

INJURY FROM FIRE.

Since the box is rarely more than 12 inches from the ground, it is within easy reach of ground fires. As both box and face are saturated with resin, a fire once started in the box may burn the tree off at the base or render the face and box unfit to produce gum. In cupped timber the cups are moved up at the end of the season and are less exposed to fires.

Another source of fire arises from the resin which impregnates the ground at the base of the tree. Such resin may come from losses in dipping, overflow from boxes on very productive trees, or from leaning trees. This waste resin may defeat the entire purpose of raking. Spilling is less likely to occur with cups than with boxes, since the former can be detached and held directly over the bucket in dipping. By having extra cups for very productive trees the chipper who visits them weekly can quickly change the full cups for empty ones and thus prevent overflow.

The immediate danger of destruction of the timber is not so great while the trees are being turpented as when the crop has been abandoned, since during the period of active operation the trash is raked from about the trees. Few tracts of timber escape the annual burning over, and turpented trees are often either killed directly or permanently injured. While the damage to abandoned cupped timber is heavy, it is not so serious as in boxed timber, since the cups are removed when the trees are abandoned and the faces are the only source of fire risk.

INJURY TO GROWTH.

The box enters the tree so deeply as to injure its vitality and retard the process of growth. Boxes are generally cut in the most prominent root swellings, especially in leaning trees, as when so placed they will more readily catch the gum and are also easier to cut. The box undoubtedly curtails the food supply of the tree to a considerable extent and accounts for the fact that more "boxed" than "cupped" trees die after tapping.

The box weakens the tree so that it is liable to be blown down by the first storm. This is especially true of small timber which may have from one-half to two-thirds of its diameter severed by the box, and of timber in old orchards that has been "back-boxed," i. e., boxes cut between the old ones wherever there is available space.

The following tabulation compares the number of dead and blown-down trees in half crops worked with cups and boxes for one season:

	Trees blown down.		Trees dead.	
	Boxed.	Cupped.	Boxed.	Cupped.
After 16 chippings.....	5	1	2	1
After 32 chippings.....	8	3	35	16

Since the box fills with water after the trees are abandoned, the surrounding wood is kept moist, increasing the likelihood of attack by fungi and subsequent decay. In some cases the box is filled with earth after abandonment to prevent it from catching fire. While it may serve the latter purpose, the procedure is scarcely to be recommended, since the earth retards evaporation of the water and hastens decay.

Trees that have been "boxed" are sometimes attacked by bark-boring and wood-boring insects, the former killing the trees and the latter seriously damaging the wood.¹

QUALITY OF LUMBER IN "TURPENTINED" AND "ROUND" TIMBER.

The wood back of the "faces" in timber that has been turpented for several years is generally impregnated with resin for a depth of from one-half to one and one-half inches. As very resinous material

¹ See U. S. Department of Agriculture Farmers' Bulletin 476, and Yearbook 1909, pp. 410-412.

will not make high-grade lumber, the proportion of high-grade material that can be cut from "turpented" timber is somewhat less than in the case of similar "round" timber. However, in many cases the process of squaring up the log by sawing off slabs will remove the resinous parts, and the grade of the boards finally cut will not be affected. Tests have shown that the strength of the wood is not altered by turpentineing.

QUALITY OF GUM FROM BOXED AND CUPPED TIMBER.

As the height of the face increases, the distance the resin must flow to reach the box increases correspondingly. During its journey the gum is constantly losing turpentine by evaporation. Thus, the percentage of turpentine in the dip decreases each year boxed timber is tapped, while the amount of scrape increases. Cups are designed to be raised each season, and thus the gum has to flow a comparatively short distance.

The resin acids in the crude gum readily absorb oxygen, which darkens the rosin. The higher the face the longer the gum is subjected to atmospheric oxygen, so that, with boxed timber, light rosins can be obtained only during the first two years. Another factor which produces dark-colored rosin is the gum that remains attached to the face after the period of collection has passed. This gum becomes yellow to dark brown, and as the following year's gum flows over it to the box, a certain amount of this highly colored product is always dissolved, so that when ordinary methods are used only the lower grades of rosin are produced from gum coming from five-year boxes. In raised cups the gum flows only over the face made during a single season. In practice, however, the cups are seldom raised after the third year, since this greatly increases the cost of collecting the gum.

COMMERCIAL DISTILLATION OF CRUDE GUM.

The apparatus commonly used in the United States for distilling gum consists of the simplest type of still, with a "worm" for condensing the vapors (Pl. VIII and fig. 5). A shed, generally open on all sides, covers the still proper, and another and smaller building, placed a short distance away as a precaution against fire, is used for storing the turpentine. It also contains the kettle for heating glue to coat the inside of the turpentine barrels. In many cases the still and warehouse are under one roof. A charging platform is built flush with the collar of the still, the barrels of gum being rolled upon it by means of skids.

The capacity of stills varies from 10 to 40 barrels. Fifteen and twenty barrel stills are the most common. The term "20-barrel still" refers to the total capacity of the still and not to the number of barrels of gum in a charge. The size of the latter is determined by

the nature of the gum; the older the gum the smaller the charge. Even with "virgin dip" the still is only filled to three-fourths of its capacity, while with dip and scrape from four or five year "boxes," which foams considerably, only about one-third the capacity of the still is used. If the material rises into the still head there is danger of it forcing an exit between collar and still head and setting fire to the platform.

The still body is about two-thirds as high as wide, with a rounded top and a slightly concave bottom, the latter permitting the rosin to

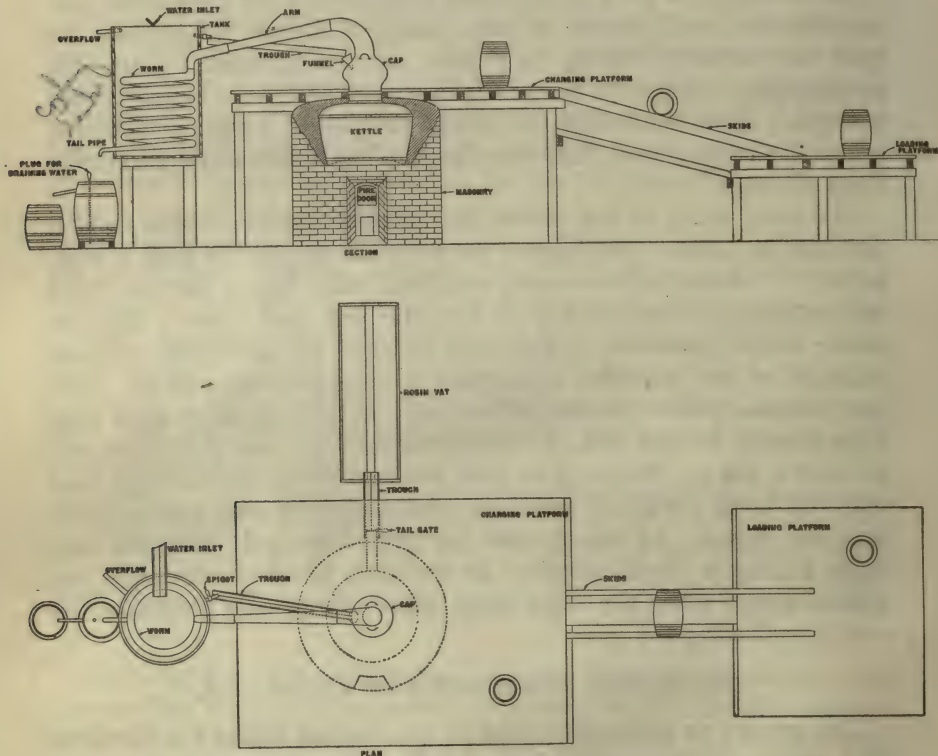


FIG. 5.—Arrangement of apparatus in turpentine still.

drain thoroughly. The still head is generally spherical¹ and is connected with the worm by two sections of pipe called the "arm" and "gooseneck." The worm makes about $6\frac{1}{2}$ turns in a wooden tank holding the condensing water, leaving the tank by means of a short pipe called the tailpiece. The entire apparatus is made of sheet copper. For a 20-barrel still, the side, top ("breast"), and collar of the still proper are made of 14-gauge copper, the rosin spout of 11-gauge, and the bottom of 4-gauge copper. The worm and connecting pipes are made of 18-gauge copper.

¹ Several forms of still head are in use.

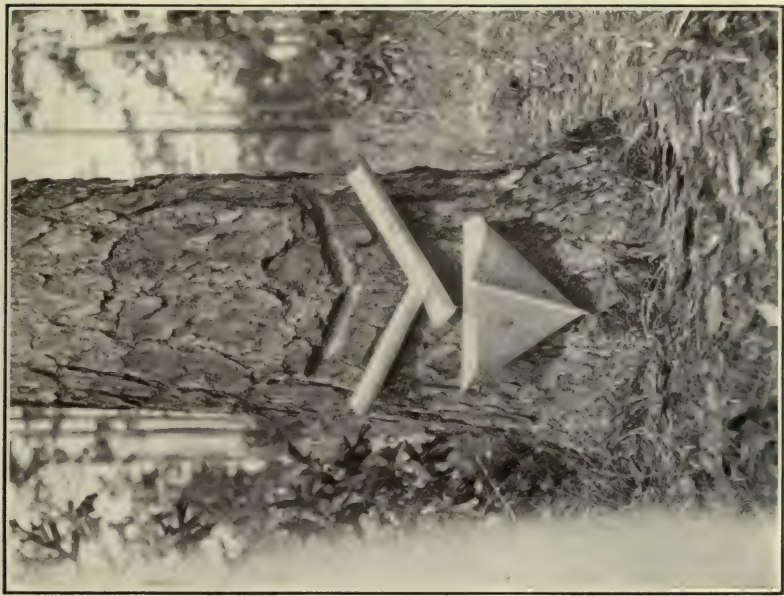


FIG. 2.—ADJUSTABLE GUTTER.



FIG. 1.—CUP WITHOUT GUTTERS OR APRON. SECOND YEAR
FACE.



TURPENTINE STILL SHOWING CONDENSER.

Glue vat for coating inside of turpentine barrels on extreme right.

DISTILLATION PROCEDURE.

The still is charged by removing the still head and gooseneck and dumping in the gum from the barrels. After most of the dip has run from the barrels they are thoroughly drained over a special trough. It is hard to remove the dip from the barrels during cold weather, and distillation is not carried on extensively in winter. When scrape is distilled alone, and the still is hot from the previous run, it is customary to pour in 5 or 6 buckets of water or a couple of barrels of dip to cool off the still and prevent the first scrape put in from "burning."

After the still has been charged the cap is put on and connected with the worm by means of the gooseneck. The joints are then luted with clay. The fire is started under the still, and its intensity regulated solely by the peculiar noise made by the gum during distillation. Crude gum always contains a certain amount of water (from 5 to 10 per cent), and since the gum melts rapidly, a mixture of oil and water soon appears at the end of the worm. A distillation requires from 2 to 2½ hours; all the water originally present distills over during the first one-half to three-fourths of an hour. The "stiller" follows the course of the distillation by placing his ear near the lower end of the worm, where the characteristic sounds made by the boiling gum are most audible, and by examining portions of the distillate collected in an ordinary drinking glass and noting the proportions of water and turpentine. The point at which additional quantities of water should be added is indicated by a peculiar strident sound, characterized as the "call for water."

The water added is obtained from the top of the cooling tank. It flows from a cock, by which the size of the stream is regulated, by way of a trough into the still through a funnel placed in an opening in the cap. The water at the top of the tank is always warm, and often very hot. Some distillers obtain this water from the bottom of the tank, claiming that the distillation is easier to regulate with cold water. About 2½ barrels of water are run in for each distillation, the amount varying with the size of the charge.

The critical period during distillation is passed when all the water in the gum has been driven over, since as the water is vaporized it swells the viscous gum to such an extent that it may overflow into the worm or escape through the joints, provided sufficient space has not been left in the still for this expansion. "Dip" and "scrape" from high faces are especially likely to boil over. The tendency to foam over is indicated by the sound of tumultuous boiling at the end of the worm. When this occurs the fire is urged as rapidly as possible, the resinous chips obtained by skimming the gum usually being added, and the increased temperature maintained until the

gum returns to a normal state of distillation. The heat is increased because at the temperature at which the water is vaporized, the gum is still in a viscous condition, and is so tenacious as to form a mass of bubbles whose shells are not ruptured by the inclosed steam; hence the gum swells enormously. In order to remove this water, it is necessary to heat the gum to so fluid a state that the bubbles will burst readily and allow the steam to escape. After this inclosed water has been driven off the distillation usually runs along smoothly, since the water that is added merely comes in contact with the surface of the gum. If excessive foaming takes place after the stream of water has been started, the fire is urged as usual, but the flow of water is not diminished. If the gum should boil over the distillation is spoiled, and must be repeated.

The end of the distillation is reached when a portion of the distillate collected in the test glass shows only a very small proportion of turpentine. All the turpentine is seldom removed, if the distiller wishes to obtain a high-grade rosin. The stream of water is now cut off, and the fire extinguished with water to prevent the rosin from igniting when it is run out, and to prevent scorching the small amount of rosin that always adheres to the bottom of the still.

Skimming.—The gum always contains more or less trash, such as sand, chips, needles, pieces of bark, etc. This all goes into the still along with the gum, and is removed at various stages during the distillation. The chips are removed with a skimmer, 16 inches long by 14 inches wide, made of wire netting and attached to a long handle.

Except in the case of dip collected at the end of the season, skimming is done as soon as the charge is fluid. In other cases skimming is done either at the point when the water originally present in the gum has passed over, or at the end of the distillation when the rosin is ready to be run off. When the gum contains a considerable amount of trash, especially bark and needles, a lighter resin will be obtained by skimming before distillation. However, in the case of "old stuff," there is considerable difficulty in getting the charge fluid enough for skimming without excessive loss of turpentine and the danger of foaming.

TREATMENT OF THE ROSIN.

After the distillation is ended, the rosin, at a temperature of 302° to 392° F., is run out by means of a pipe extending flush from the bottom of the still and closed by a gate valve. Usually the rosin flows through a set of four screens into a vat sunk into the ground. One, two, or three screens may be used, however, instead of four. The vat is about 4 by 15 feet at the bottom, 4½ by 15 feet at the top, and 2½ feet deep. The screens are sufficiently large to cover it, with the exception of the top one, which is only half the length of the still, and is intended to catch only the coarsest chips. The top screen is

from 6 to 8 mesh, the second 14 mesh, the third 32 mesh, and the bottom 60 mesh. The bottom screen is covered with a layer of cotton batting to remove the finer particles of dirt.

The rosin remains in the vat from a few minutes to an hour, according to the temperature at which it left the still. It is next dipped into crude barrels made on the spot, holding about 450 pounds net. If dipped while too hot and fluid, considerable leakage occurs between the staves, which may in a measure be prevented by luting with clay. The rosin requires about 24 hours to become solid.

The cotton batting, after being used to strain the rosin, is known as "batting dross" or "rosin dross." As cotton is very absorptive, a large amount of rosin is retained. Recent analyses made by the Forest Service indicated that rosin dross contains from 75 to 90 per cent by weight of rosin.

It has been the practice to burn under the still a certain portion of the chips removed by skimming and in the screens, and to throw away the rest. In this way, piles of discarded chips often grew to large size before the stills were moved. Such piles, of course, contain considerable rosin, and during 1911 and 1912, owing to the high price of naval stores, operators found it profitable to sell not only the dross, but the skimmings and similar material to extraction plants.

TREATMENT OF THE TURPENTINE.

The distillate issuing from the worm, and consisting of a mixture of water and turpentine, runs into an ordinary 50-gallon barrel, where the separation of the water and turpentine takes place by gravity; the turpentine, being lighter, floats on the top. The bottom of this barrel contains an opening, closed with a long wooden plug, by which the excess water is allowed to escape as the volume of the distillate increases. In most cases a second container, consisting of a barrel whose upper half has been sawed off, receives the turpentine flowing from the top of the first barrel through a short pipe, to permit of more perfect separation. A thin yellow scum forms the line of demarcation between the water and turpentine. The latter is dipped out carefully and poured directly into the barrels in which it is sent to market.

The first runnings of turpentine are colored more or less green with copper salts, due to the action of acetic and resin acids on the copper of the worm and still. The green color is especially noticeable when the still is first used after a period of idleness. When the still is in continuous use the color in the first runnings is very slight.

The turpentine barrels must be thoroughly tight. They are usually made of sound white oak, thoroughly driven, and coated on the inside with glue. Each barrel holds about 50 gallons, some space being left for expansion of the contents.

FRENCH METHODS OF COLLECTING GUM.

MANAGEMENT OF FORESTS.

A large proportion of the French forests exploited for resin are situated along the coast, where the shifting sand dunes have been planted with maritime pine (*Pinus maritima*).

The forest rotation varies from 60 to 75 years, and since maritime pine is a prolific seeder, a new growth readily springs up on cut-over areas. At the end of 10 years the stand is thinned, and thereafter at 5-year intervals. During the first thinning the lower branches of the trees that are left are lopped off to a height sufficient to insure a clean bole for turpentine during subsequent years. The wood and resin rights are sold for a period of five years, the turpentine being done by the purchaser, who, at the end of the operation, fells those trees marked for thinning.

The wood is used for mine timbers, boxes, crossties, telegraph poles, etc. Turpentine timber is preferred over unturpentine, since it is very resinous and so resists decay for a longer time. The last thinning is made when the trees are about 30 years old, and the remaining pines, numbering about 50 per acre, are then turpentine. At the end of the rotation period, owing to various casualties, there remain only about 30 trees per acre for lumbering.

Up to the year 1860, practically all resin was collected in holes scooped out of the sand at the base of the trees. This method was wasteful, and the gum was badly contaminated with sand and other debris. The use of an earthen pot with a gutter was suggested in 1840 by M. Hugues, of Tarnos, but was not taken up until 1860. At present the Hugues cup and gutter system has almost entirely superseded the old method.

BARKING.

Before the turpentine season opens the outer bark is removed over an area exceeding somewhat the area to be chipped during the coming season. The denuded area measures 24 inches in height and 6 to 8 inches in width, and in depth reaches to the living layer of the bark. The operation is performed carefully with a wide-bladed ax. Above the height of a man the ax is replaced by a tool with a hook-shaped blade, 3 inches wide, attached to a long handle, which is wielded with a pulling motion. The bark is removed to prevent dulling the delicate edge of the chipping tools, and the rays of the sun on the exposed area are supposed to have a beneficial effect in stimulating the flow of resin.

HANGING CUPS (HUGUES SYSTEM).

The face is opened the first of March by removing a chip 1.6 inches high, 3.5 inches wide, and 0.4 inch deep from near the base of the tree. The tool used is a peculiarly shaped instrument called the "abschot" (Plate IX-3). A zinc gutter is inserted at the base of the wound in a



FRENCH TURPENTINE TOOLS.

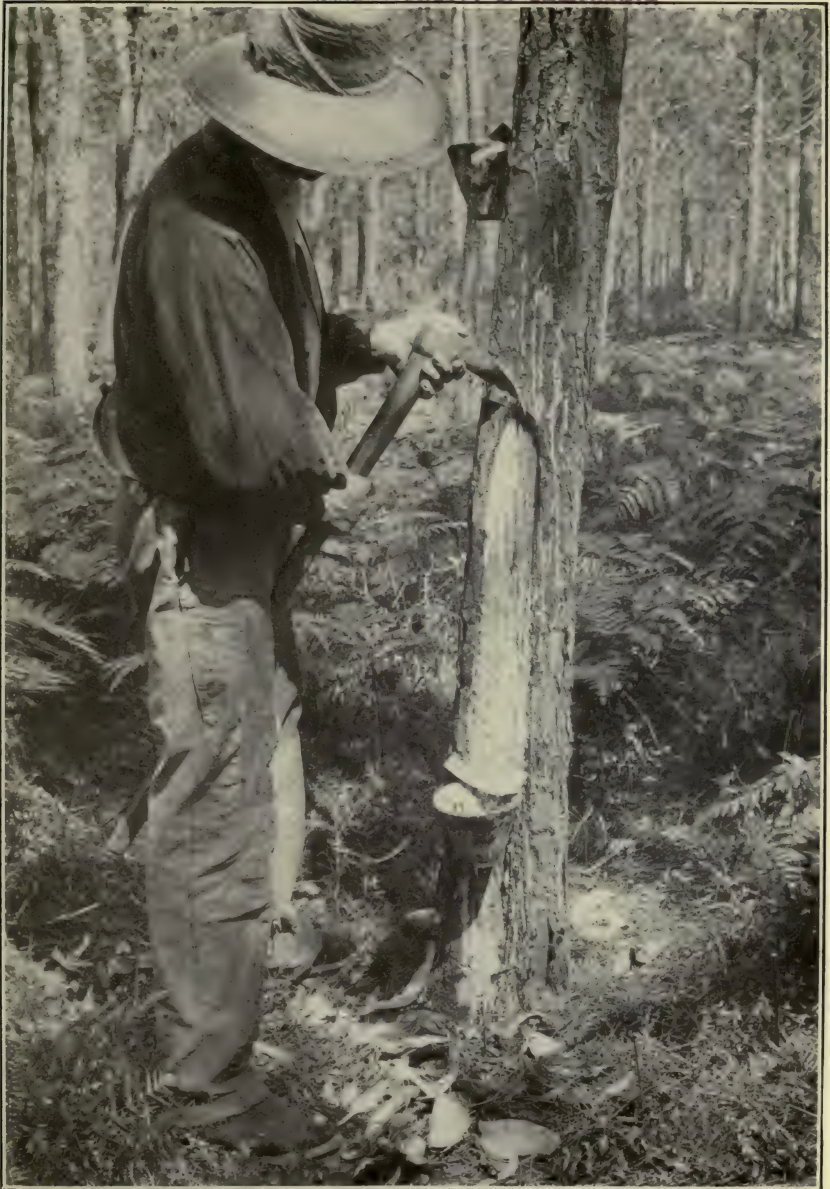
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PLATE X.



CHIPPING A LOW FACE, SHOWING THE SHAPE OF THE AXE AND RELATIVE SIZE OF THE FACE.



CHIPPING A HIGH FACE, SHOWING THE PRACTICE OF INSERTING CHIPS ON SIDES OF FACE ON LEANING TREES TO GUIDE GUM INTO CUP.

gash made by a chisel (Plate IX-5) with a cutting edge in the shape of the arc of a circle. Below the cutting edge is a socket into which the gutter fits. The gutters are 8 inches long and 2 inches wide, and in some cases are provided with five teeth. A gash 0.2 inch deep has been found sufficient to hold the gutters, since the resin soon enters the wound and acts as a cement.

The pots used are made of glazed earthenware, conical in shape, 4½ inches wide at the top, 3 inches at the bottom, 6 inches high, with a capacity of about 1 quart (1 liter). The cup rests on the ground the first year, and is raised along with the gutter at the beginning of each succeeding year of operation. The upper part of the pot is held in place by the gutter, which projects out and downward, while the base rests on a nail driven into the tree. The top of the cup is never provided with a hole for hanging on a nail.

CHIPPING.

Chipping is begun about the first of March and ends the latter part of October. A total of 40 chippings is usually made in one season.

The chipping tool (abschot) is of two types: One type is a combined adz and gouge, the blade hanging at right angles to the handle, its edge shaped like the arc of a circle. In the other type the blade is parallel to the handle, but bent outward so that the cutting edge does not fall within the plane of the handle.

In using the abschot the workman stands to one side and in front of the face, with the handle between his legs (Plate X). In removing the chip the blade is inserted at the extreme upper corner of the face and is drawn diagonally toward the workman. Little effort but great skill is required.

The wood removed in chipping is in the form of shavings, so that the edges of the face are perfectly smooth, allowing the wound to heal rapidly. The face increases in height about 0.6 inch at each chipping. After the face is opened, chipping is repeated every 8 days from March to May, every 5 days from June to the end of August, and every 8 days from September to the middle of October or the first of November.

After the face has reached the height of a man the abschot is discarded for the rasclet. The rasclet (Plate IX-4) has a hook-shaped blade, with its edge at right angles to the long handle, and the chip is removed by a pulling motion. In the case of leaning pines, wooden chips are inserted along the edge of the face to guide the resin toward the pot (Plate XI).

The dimensions of the faces must conform to the tapping specification, and frequent inspection is made by government officials to see that these are carried out. Since the wages of the workman consist of half the proceeds from the sale of the resin, he naturally wishes to collect as much resin as possible, and is tempted to increase the size

of the face beyond the prescribed limits. An instrument devised by Demorlaine in 1898, called the "facemeter" (quarrimetre), permits of a ready and accurate measurement of the face.

SYSTEMS OF TURPENTINING.

Two systems of turpentineing are employed, "*gemma a vie*" and "*gemma a mort*." "*Gemma a vie*" is the system used when the tree is to be moderately turpentineed for a long period of years; "*gemma a mort*" when the trees marked for thinning or final felling are to be made to yield the greatest possible amount of resin in five years without actually being killed.

The following articles are taken from the turpentineing specifications issued by the French Government in 1909:

Gemma a vie.—The tapping will take place on either one or two faces, according to the indications of the Service des Eaux et Forêts. Only those trees can be tapped with two faces which have been designated for this purpose.

The faces will be started above the swelling of the root and raised either vertically or in accordance with the grain of the wood.

If the tapping period is for five years, the face can be raised 0.60 m. (24 inches) during the first year and 0.65 m. (26 inches) during the following years in such a manner that the total height does not exceed 3.2 m. (10 feet 6 inches).

If the tapping period is for four years the face can be raised 0.60 m. (24 inches) during the first year; 0.65 m. (26 inches) during the second; 0.85 (33 inches) during the third, and 1 m. (3 feet 3 inches), in such a manner that the total height does not exceed 3.1 m. (10 feet 2 inches).

In every case the width of the faces should not exceed 0.09 m. (3.5 inches) the first year, 0.08 m. (3.1 inches) the second, 0.07 m. (2.8 inches) the third, and 0.06 m. (2.4 inches) at the beginning of the fourth.

The decrease in width should take place progressively in such a manner that the width of the face at the end of one year shall be that at the beginning of the year following.

The depth should not exceed 0.01 m. (0.4 inch), the measure being taken under a cord stretched from one border of the face to the other, at the beginning of the red part of the bark.

The tapping will take place according to the directions of the Service des Eaux et Forêts; either by fours (au quart), the faces up to the fourth inclusive being made, as far as possible, two by two at the extremities of the same diameter; or by threes (au tiers), the faces up to the third inclusive being made by dividing the circumference of the tree into three nearly equal parts; the second should be opened at the right of the first when facing the latter.

In case of the absence of directions in the contract the tapping will be done by fours.

Gemma a mort.—If the trees to be tapped *à mort* form part of the sale or are abandoned to the lessors, the latter can work them as they think best.

In the contrary case the lessors should not tap them in a manner to diminish the value which they should have as fuel and structural timber. The dimensions of the face should be such that its whole area never exceeds the limits of an ordinary face.

The tapping operation will be confined between March 1 and October 31 of each year, but the contractor can commence to bark the pines which are to be tapped and place the gutters February 1.

He can also collect the scrape up to December 1 of each year of the tapping period, except the last year, when this operation should be ended the 15th of November.

Two kinds of scrape are distinguished in France. The hardest kind adhering firmly to the face is called "barras," while the soft scrape is called "galipot." The tool used (Plate IX-1) for removing the scrape resembles that used in removing the outer bark, except that the blade is only $1\frac{1}{2}$ inches wide. Usually the trees are scraped but once a season, in November, but sometimes an additional scraping is made in June.

The method of collecting the crude gum is practically identical with that used in America.¹ The resin is temporarily stored in wooden tanks sunk into the ground at convenient points in the forest until it is ready to be transported to the still.

FRENCH DISTILLATION METHODS.

In French operations the barrels of resin as they come from the forest are usually stored in large tanks, so as to form a reserve supply to be worked up during the winter months when no resin is collected. The storage tanks are sunk into the ground at a distance of 75 feet from the still, and the resin transferred to the latter by means of an overhead trolley. The tanks are built of brick or cement and covered with tile.

PURIFICATION OF THE RESIN.

The resin is often subjected to preliminary treatment previous to distillation to remove the trash. This is not done, however, unless high grade rosin can be produced which will bring a good price, since the process results in loss of turpentine and requires extra fuel and labor.

The preliminary treatment involves fusion, clarification, decantation, and straining. The fusion is performed in open or closed pans.

Open pans.—The resin is liquified in a cylindrical copper pan 6 feet in diameter and $1\frac{1}{2}$ feet in depth, with a slightly concave bottom.

¹ Comparison of yields of crude gum per inch of width of face, French and American methods:

Data from French operations indicate an average yield of 1.8 liters of crude gum per face per year, or 4 pounds.

If chipped 40 times yield per face per chipping=0.1 pound.

If face is 3.5 inches wide yield per inch width=0.029 pound.

If face is 4.0 inches wide yield per inch width=0.025 pound.

Data from American operations:

Crop A—8,000 faces. (See page 25):

206,235 pounds gum in 4 years=6.4 pounds per face per year.

43,633 pounds scrape in 4 years=1.4 pounds per face per year.

Total..... 7.8

If chipped 32 times yield per face per chipping=0.244 pound.

If face is 12 inches wide yield per inch width=0.020 pound.

If face is 14 inches wide yield per inch width=0.017 pound.

Crop D—8,000 faces. (See page 25):

279,260 pounds gum in 4 years=8.7 pounds per face per year.

53,915 pounds scrape in 4 years=1.7 pounds per face per year.

Total..... 10.4

If chipped 32 times yield per face per chipping=0.325 pound.

If face is 12 inches wide yield per inch width=0.027 pound.

If face is 14 inches wide yield per inch width=0.025 pound.

This pan, inclosed by brickwork and heated by the flames from a fire-box, has a capacity of about 325 gallons.

After the pan has been filled with crude resin the heating is conducted very slowly to prevent "burning." A workman stirs the mass constantly with a wooden paddle to render the heating uniform. When boiling begins the heating is discontinued, as the temperature should never be allowed to exceed 90° to 100° C. To clarify the mass the temperature is suddenly reduced by drawing the fire. Sometimes a small amount of water is thrown into the fire box and upon the bottom of the pan. If this delicate operation is successful, the mass will settle into several layers after standing for four or five hours. A layer of chips, bark, etc., will be found on the surface; then a layer of resin; and below this a layer of colored water with a deposit of sand, etc., on the bottom. The floating chips and bark are removed by skimming. The resin may then be run out through pipes arranged at different levels, but usually is dipped out. The heaviest material is filtered through a screen to remove the sand, the filtrate separating into a layer of colored water surmounted by a small amount of inferior resin. By this procedure a resin is obtained which on distillation will yield a rosin several grades higher than the original resin would give if distilled with all its impurities.

Purification in an open pan results in the loss of from 2 to 3 per cent of turpentine, and there is considerable danger of fire. Several pans provided with covers have been designed to overcome the objections cited.

Closed pans.—The pan designed by Dromart (fig. 6) illustrates the closed type. It is provided with a horizontal cover whose edge fits into the groove (R), the latter being fed by a stream of cold water so that the cover is hermetically sealed. The pan is charged without loss of turpentine by means of a box (B) with a trap in the bottom worked by a lever. By manipulating this lever the resin contained in the box falls into the pan. To obtain uniform heating the resin is stirred by means of an agitator (G). At the end of 4 to 5 hours the melted resin shows a temperature of from 85° to 90° C., and a jet of steam issues from a test hole in the cover. The heat is then reduced, and the liquid mass cooled by dumping in one or two boxes of resin through the trap. After stirring vigorously the mass is allowed to rest for 12 hours. The resin is then decanted through a pipe (V) situated above the bottom so as to keep the layer of water and dirt below its orifice.

It is sometimes difficult to separate the water and solid impurities from the resin, owing to the fact that the density of the resin and that of the water are so nearly the same. The density of a gum containing 80 per cent rosin and 20 per cent turpentine, at 20° C., is about 1.023, while at the same temperature distilled water has a density of 0.998. Since the water in the resin contains certain amounts

of dissolved matter, its density may be greater than the figure given, while the density of resin richer in turpentine will be less than 1.023. To separate the two it is necessary to lower the density of the resin or increase the density of the water. The former is accomplished by adding certain amounts of "heads" and "tails" from a previous dis-

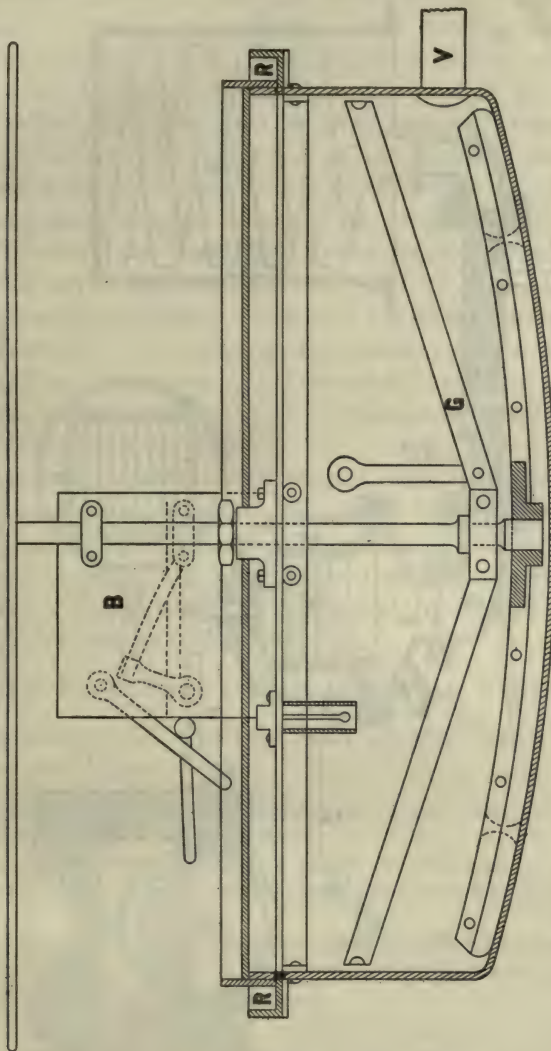


FIG. 6.—Type of closed evaporating pan used in France to prevent loss of turpentine when gum is purified.

tillation, and the latter by adding a cheap salt, such as common salt or soda.

DISTILLATION BY DIRECT HEAT.

Direct heating is the method of distillation generally employed in France. The type of still and its method of operation are exactly the same as in the United States.

DISTILLATION BY STEAM.

Of the 200 stills producing turpentine and rosin in France, only about 30 use steam. Several types of steam apparatus are employed, but the Dorian still will serve as an example of the stills heated by steam only.

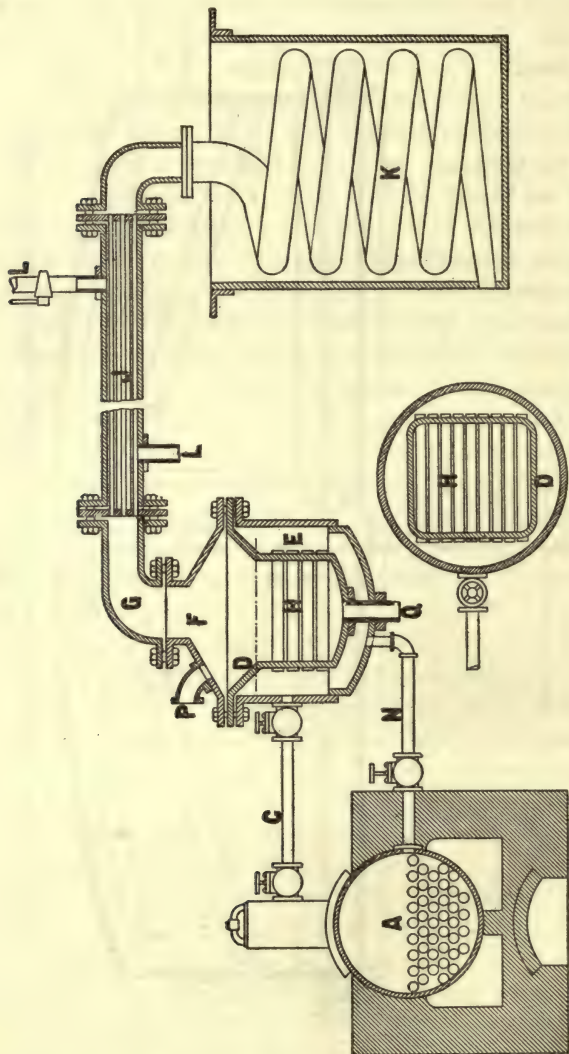


FIG. 7.—Type of steam turpentine still used in France.

This still is remarkable in its simplicity, compactness, and efficiency. Its principal advantage lies in the fuel economy obtained by returning directly to the boiler the condensed water obtained from the steam used in heating the still, instead of allowing it to flow away as a complete loss or running it into a tank for feeding the boilers.

The apparatus (fig. 7) consists of a steam generator, still, and condenser. The generator A, which may be of any type, feeds the

still by a pipe C attached to the steam dome. The still is constructed entirely of steel made to stand a pressure of 10 atmospheres, although 6 atmospheres is never exceeded in actual practice. The still consists of a gate valve P for introducing the crude resin and another gate valve Q for discharging the rosin; the body of still D, shaped like a prism; a steel jacket E, circular in shape; 32 tubes H, arranged crosswise in four series of eight tubes each, running through the still from one side to the other, through which passes steam from the jacket E; a hood F; a still-head G; and a pipe N, which leads back to the boiler the water produced by condensation of steam in the still. Since the pressure in the boiler and in the steam jacket is the same, this condensed water readily returns to the boiler by its own gravity.

The crude resin, divided into thin layers by the pipes H, distills rapidly. To carry the turpentine over, water is supplied through a funnel in the top of the hood F.

The condensing apparatus consists of a tubular condenser J and an ordinary worm K. The condenser is made up of a series of tubes from 10 to 12 feet long, riveted to two steel plates, and is fed with cold water through LL'. The tubular condenser is so efficient that the worm becomes almost useless.

A still having a capacity of 92 gallons (about 2 barrels) has a heating surface amounting to about 120 square feet, and permits the distillation of one charge in about 40 minutes.

When the turpentine is completely removed the introduction of water ceases. The heating is continued by means of the steam jacket until the rosin is free from water.

COMPARISON BETWEEN DIRECT AND STEAM HEATED STILLs.

The disadvantages of distilling with directly heated stills may be summarized as follows:

1. During the distillation of the crude resin the ligneous impurities may undergo a partial carbonization which colors the rosin.
2. The rosin becomes exceedingly dark at high temperatures.
3. The rosin may undergo incipient decomposition and color the turpentine more or less yellow.
4. Distillation by direct heat is a delicate operation, difficult to regulate, and requiring an experienced man. It can be applied only to a comparatively small quantity of material, if good results are to be obtained.
5. Increased fire risk.

By using stills heated by steam the above disadvantages are removed, but others are introduced. The reasons why the use of steam stills has not become general are:

1. Complicated apparatus.
2. Greatly increased cost of apparatus and expense of operation.
3. Necessity for a large stock of crude resin, if the still is to be operated economically.
4. With the majority of apparatus the crude resin must be given a preliminary treatment to remove chips, bark, sand, and other trash.

5. Only a slight increase in the commercial value of the turpentine and sometimes of the rosin.¹

However, when fuel and crude resin are plentiful, steam stills have the following important advantages:

1. Very pure turpentine, better than that obtained by direct heat.
2. Rosin not superheated, and but slightly colored.
3. Simple and easy control of the distillation.
4. Decrease of fire risk.

As a general rule, an experienced distiller will obtain as good results with the ordinary American still as with a steam still.

THE SUPPLY OF LONGLEAF PINE FOR TURPENTINE OPERATIONS.

Up to the middle nineties the large supply of yellow pine stumpage, the prejudice against lumber cut from turpented trees, and the lack of adequate transportation facilities in many regions where turpentine operations were conducted, caused large bodies of turpented timber to be abandoned and left to be destroyed by fire, wind, and decay. It is estimated that in each of the States of North Carolina, South Carolina, Georgia, Florida, Alabama, and Mississippi the loss in boxed timber has amounted to from three to ten billion board feet.

At present the damage to standing timber due to turpentine operations has been considerably reduced. The cup systems lessen the fire risk and the heavy demand for lumber, coupled with improved transportation facilities, has shortened the period between the end of turpentine operations and the beginning of lumbering. However, as the supply of timber available for turpentine has grown smaller, the practice of turpentine undersized trees has become common, especially in second-growth stands that have come up after old lumber operations. When a tree under 6 inches in diameter is boxed it seldom makes further growth, and cupping has almost as bad an effect. Not only is further growth prevented, but the tree becomes a menace to the rest of the stand through windfall, fire, or decay. The future production of naval stores in the Southeast is rendered uncertain by the practice of turpentine small trees, and the future supply of longleaf pine is endangered. Moreover, the returns derived from turpentine small timber are, as a rule, hardly sufficient to cover the expense of operation.

The scarcity of longleaf pine suitable for turpentine has reached an acute stage in North Carolina, South Carolina, and Georgia, and is the natural result of the exhaustion of the virgin pine forests. While considerable "round" timber—that is, timber which has never been tapped—remains in Alabama, Mississippi, Louisiana, and Texas, it is

¹ On Apr. 5, 1911, the quotation at Savannah on "B" rosin, the lowest grade, was \$8.15 per barrel, while "WW," or the highest grade, brought but \$8.62 per barrel. The average price of "WW" rosin during the naval stores year 1913-14, was \$6.38, and of "B" rosin, \$3.96 per barrel.

for the most part held by large lumber syndicates, which usually are unwilling to permit turpentine operations. A few of the holding companies are beginning to permit turpentine, either carrying on the operations themselves or leasing the privileges to large naval stores companies. In Florida the small operator has more oppor-

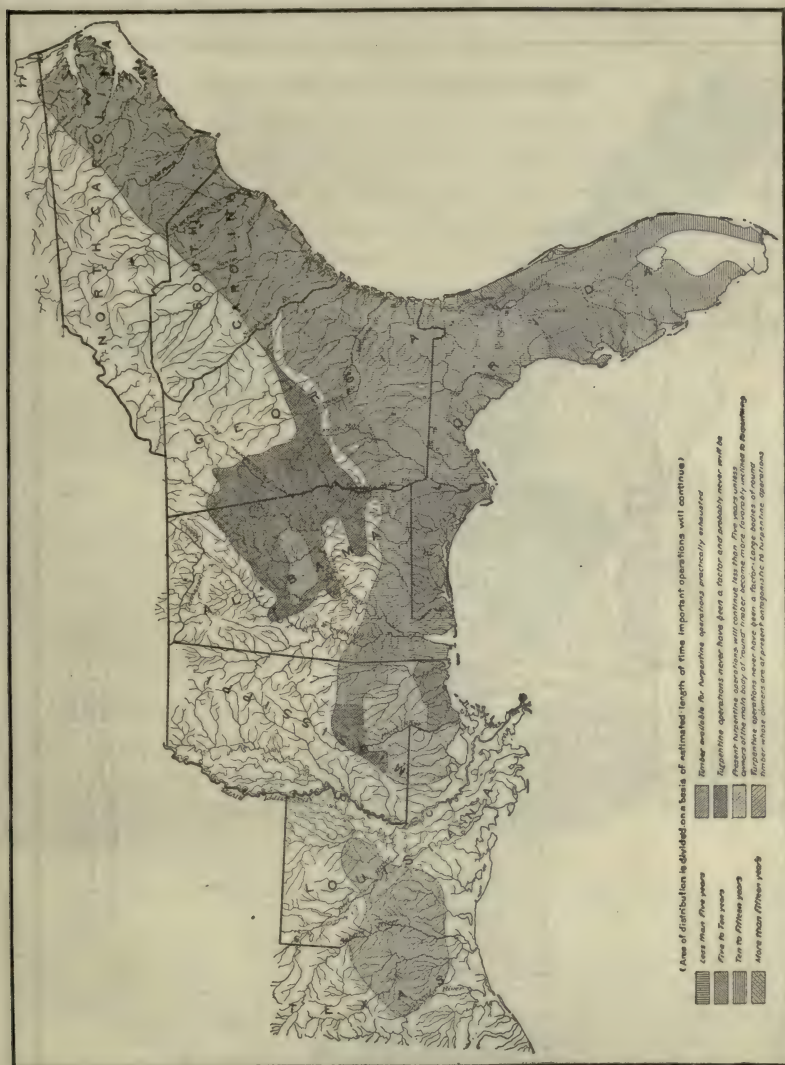


FIG. 8.—Approximate distribution of longleaf pine with reference to turpentine operations.

tunity, since much of the timber is at present in the hands of turpentine men.

Figure 8¹ shows the distribution of longleaf pine. The various styles of crosshatching denote the estimated number of years (from 1909) for which virgin timber will be available for turpentine opera-

¹ Taken from unpublished report, "Investigation of the Naval Stores Industry," by A. L. Brower and J. D. La Fontissee (1909).

tions. By this it is not meant that production will cease in any given region within the time indicated on the map, but that after the lapse of such time it is probable that the turpentine operator will have to make use of second-growth timber and that left by the lumberman. Under these conditions the production will amount to but a fraction of the present production. This prediction is based on the supply of

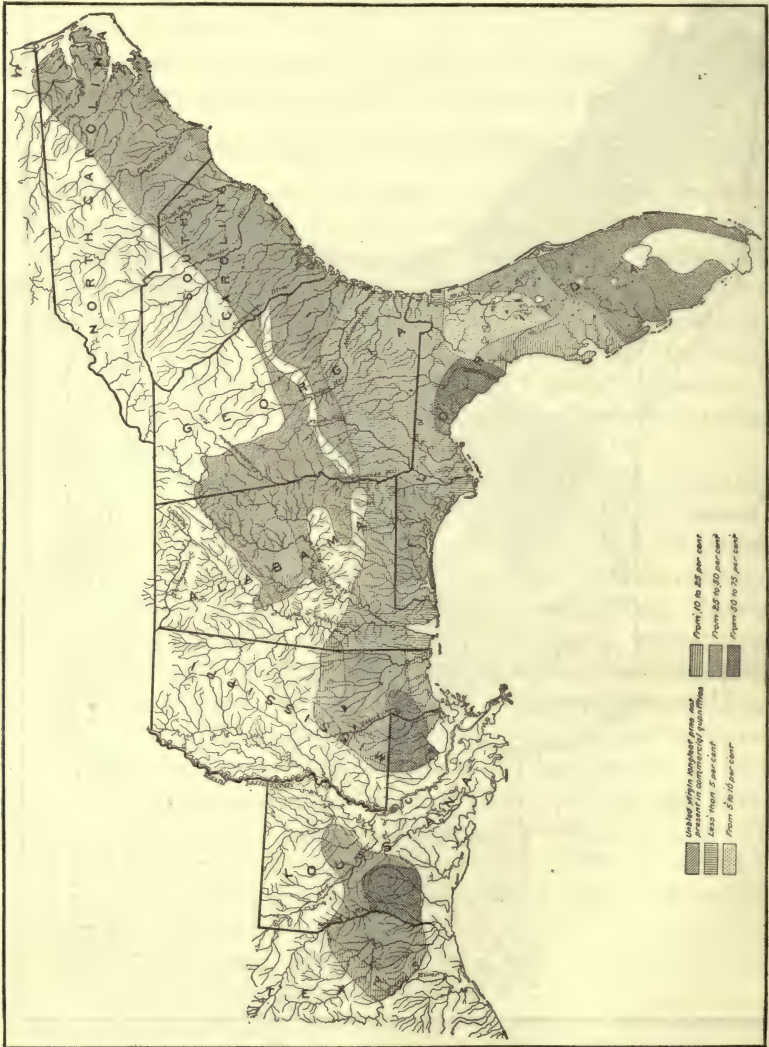


FIG. 9.—Approximate distribution of longleaf pine with reference to percentage of original stand remaining unbled.

longleaf, and does not take into account the possibility of utilizing loblolly and shortleaf pine as a source of naval stores.

Figure 9¹ indicates the percentage of the original longleaf pine that still remained standing "round" in 1909. The data are not

¹ Taken from unpublished report, "Investigation of the Naval Stores Industry," by A. L. Brower and J. D. La Fontisse (1909).

intended to be accurate for any particular locality, but rather to represent as correctly as possible the conditions generally obtaining over the territory, the unit of consideration seldom being less than a county. The high price of naval stores in 1911 produced an unprecedented invasion of the "round" timber. It has been estimated that 75 per cent of the "round" timber held by turpentine operators was tapped in 1912.

YIELDS PER CROP IN VARIOUS STATES.

Table 16 shows the yield per crop in six States in the turpentine belt and the percentage of gum secured by boxing and cupping. It is noticeable that the States in which the yield per crop was largest also made the largest use of improved methods. The timber used in turpentine operations in Louisiana and Texas is, however, generally of larger size than in the other States mentioned, so that a somewhat larger yield per crop under the same conditions would be expected, although not such a difference as shown in Table 16.

TABLE 16.—Average yields in turpentine operations, by States,¹ during 1909.

State.	Yield of turpentine per crop.	Percentage of gum secured by cupping.	State.	Yield of turpentine per crop.	Percentage of gum secured by cupping.
	<i>Barrels.</i>			<i>Barrels.</i>	
Alabama.....	35.6	8	Louisiana.....	44.7	44
Florida.....	29.8	16	Mississippi.....	34.5	11
Georgia.....	26.5	9	Texas.....	43.5	49

¹ Taken from statistical report on naval stores by Brower and La Fontissee.

Table 17 shows the new crops started by the box and cup methods in 1909, 1908, and 1907. The figures indicate that the cup method is steadily gaining ground; they also show that North Carolina and South Carolina at present play very little part in the production of naval stores.

TABLE 17.—New crops started by box and cup methods in 1909, 1908, and 1907.¹

State.	Crops—1909.			Crops—1908.			Crops—1907.		
	Boxed.	Cupped.	Per cent cupped.	Boxed.	Cupped.	Per cent cupped.	Boxed.	Cupped.	Per cent cupped.
Alabama.....	337	131	28.0	420	84	16.7	423	71	14.4
Florida.....	1,374	326	19.2	1,593	313	16.4	2,065	210	9.2
Georgia.....	1,026	120	10.5	1,182	101	7.9	1,482	139	8.6
Louisiana and Texas.....	92	135	59.5	163	113	40.9	97	67	40.9
Mississippi.....	181	90	33.2	252	49	16.3	288	40	12.2
North Carolina.....	6			15	1	6.3	3	1	25.0
South Carolina.....	42			41			91		

¹ Taken from census report for 1909, reported as virgin, yearling, and third-year faces worked in 1909. Later statistics are, unfortunately, not available.

POSSIBILITIES OF WESTERN PINES AS A SOURCE OF NAVAL STORES.

During 1911 and 1912 the Forest Service conducted experiments on western yellow pine in Arizona, on western yellow pine, Jeffrey pine, digger pine, singleleaf piñon, and lodgepole and sugar pine in California and northeastern Oregon, and western yellow pine and piñon pine in Colorado, to determine the quantity of crude gum which could be secured from these pines by the methods ordinarily employed in the turpentineing of longleaf yellow pine in the Southeast.¹ The field work was supplemented by laboratory analyses to determine the quality of the gum.

Table 18 compares the yields obtained in Arizona with those obtained in experiments conducted on a commercial scale in Florida. The Arizona experiments show a yield from western yellow pine about four-fifths as great as that obtained from southern yellow pines on average operations in Florida in the same period of time. Weather conditions in Arizona, however, will allow only a 24, or possibly a 26-week season, as against 30 or 35 weeks in the Southeast, so that when the yields for the entire season are compared western yellow pine shows a production about two-thirds as great as that from southern yellow pine. The average proportions of rosin and turpentine in the gum were about the same in both regions, as was the composition of the turpentine.

TABLE 18.—*Comparison of yields, in pounds, of crude gum and scrape from western yellow pine in Arizona and longleaf pine in Florida.*

Locality.	Crop designation.	Total weight obtained during season.		Average per cup per week.	
		Dip.	Scrape.	Dip.	Scrape.
Arizona ¹	A	2,862.50	523.75	0.239	0.0436
Do.....	B	2,524.25	314.75	.210	.0262
Do.....	C	2,431.75	288.25	.203	.0240
Average.....		2,606.16	375.58	.217	.0313
Florida ²	A	63,615.5	9,570	.256	.0386
Do.....	B	61,161.5	7,650	.246	.0308
Do.....	C	62,587	7,245	.252	.0292
Do.....	D	73,703.5	8,880	.297	.0358
Average.....		65,266.9	8,336	.263	.0336

¹ Each crop of 500 cups chipped 24 times.

² Each crop of 8,000 cups chipped 31 times.

The California experiments on western yellow pine were carried on from July 7 to November 1, 1911, and from May 10 to August 3, 1912. The yields obtained in 1911 and 1912 are combined in Table 19, to show the flow for an entire but not a continuous season.

¹ The California and Oregon experiments were made under the direction of Mr. C. Stowell Smith and Mr. J. B. Knapp, assistant district foresters, districts 5 and 6. A complete report of this work is on file at the Forest Service, Washington, D. C., and at the Forest Products Laboratory, Madison, Wis. For a detailed description of the Arizona and Colorado experiments see Forest Service Bulletin 116, "Possibilities of Western Pines As a Source of Naval Stores," by H. S. Betts.

TABLE 19.—*Yields of California western yellow pine, by months (crop of 300 cups, chipped 28 times).*

Date of dipping.	Weight of dip.	Date of dipping.	Weight of dip.
	Pounds.		Pounds.
1912.		1911.	
May 10-11.....	175	August 16.....	217
May 23.....	153	September 5.....	189
June 6-7.....	146	September 15.....	129
June 21.....	156	September 27.....	172
July 6.....	171	October 19.....	179
July 20.....	197	November 1.....	152
August 3.....	280	November 28.....	212
Total.....	1,258	Total.....	1,250
		Total for 1912 and 1911.....	2,508
		Average per cup per week.....	0.3

The average flow per cup per week in the California experiments for a season of 29 weeks was somewhat greater than in the Florida experiments recorded in Table 18 for the same period of time. The California yield is also slightly greater when the production for the entire season is compared.

The composition of the volatile oil obtained by distilling the gum from the California trees differs from that of ordinary turpentine somewhat more than does the Arizona turpentine, but the oil probably will be satisfactory for industrial purposes. The yield from western yellow pine in northeastern Oregon was very small compared with that in California. This can be attributed in part to the unusually adverse climatic conditions during the season, but it is not likely that more favorable weather conditions would increase the yield enough to make turpentering in that region a profitable industry.

Jeffrey pine in California yielded only 61.5 per cent as much as western yellow pine farther south during the same period. The principal constituent of the oleoresin from Jeffrey pine is heptane, which can not be used as turpentine, but has been employed to some extent for medical purposes. Digger, piñon, lodgepole pine, and sugar pine in California were found to yield such small amounts of oleoresin that it would be impracticable to tap them on a commercial scale unless the particular oil they produce could be made to bring a high price for some special purpose.

Piñon pine (*Pinus edulis*) in Colorado had a rate of flow slightly over one-half that of the Florida pines for a 20-weeks' period, from June 9 to October 31. The volatile oil from the piñon gum differs somewhat from ordinary turpentine, but is probably suited for industrial use.

PROBLEMS OF COMMERCIAL DEVELOPMENT.

In considering the possibilities of commercial turpentine operations on western pines the problem of labor is one of the first that presents itself. In Arizona the Mexicans, who constitute a large part of the laboring class, are totally unfamiliar with turpentine work. Negro turpentine hands could be brought in from the Southeast, but

their transportation would be costly. A few negro hands might be secured to teach the Mexicans, but whether the results would be satisfactory is, of course, unknown. In California both Indian and white labor is available in many timbered portions that have turpentine possibilities, but here also the chippers would have to be taught how to use a hack.

The shorter season in Arizona, as compared with that of the Southeast, and the comparative severity of the winters in the timbered parts of the State, might make it necessary to discontinue operations entirely for a few months during the winter. This would necessitate the reorganization of the operating force each spring, with a great many attendant difficulties. The flow continued longer in California than in Arizona, the experimental areas in the former State showing a considerably higher average temperature than those in the latter, though the diurnal range of temperature in California was greater.

Western yellow pine timber generally grows in open stands free from underbrush, and in most cases there would be little, if any, more difficulty in moving the crude gum than in the Southeast. On rough ground burro pack trains might be used. Two small kegs or buckets, holding about 150 pounds of dip, could be slung on each animal.

The number of cups that can be hung on an acre of average western yellow pine compares favorably with the number hung on many areas now being turpented in the Southeast. The western trees are larger than most of the southeastern ones, though their bark is thicker and rougher, and the outer portion must be removed before the trees are chipped. This, of course, means the expense of an extra step not necessary in southeastern operations. Such work can be done by the use of a broadaxe or heavy spade-shaped tool with a cutting edge.

The cost of securing turpentine rights in the Southeast is constantly rising, and it is likely that turpentine stumpage could be leased at lower rates in the West. At present the turpentine and rosin used in the West is shipped from the Gulf States, and the advantage of cutting out a two or three thousand mile haul to western markets is evident.

The naval stores industry is not new in California. During the Civil War when the supply of naval stores from the South was cut off an attempt was made to supply the northern States from the Pacific coast. The industry remained active for four or five years, but suddenly declined when North Carolina again came into the market after the close of the war.

The commercial success of turpentine operations in the Southwest will be doubtful until tried on a commercial scale. Nearly as much turpentine and rosin were obtained from western yellow pine as from

longleaf, and the amount of timber available for turpentine operations in the Southeast is constantly diminishing. These two facts make it reasonable to suppose that turpentine operations in the large tracts of virgin pine timber of the West will in time be justified.

SPECIAL PROBLEMS INVESTIGATED—ARIZONA AND CALIFORNIA WESTERN YELLOW PINE.

EVAPORATION FROM CUPS.

The rate of evaporation from the cups in Arizona was determined by exposing cups half full of fresh gum to the action of the air and weighing them at regular intervals. The samples of gum were secured by taking small amounts from as many of the cups as necessary on the first or second day after a fresh streak had been put on. Two samples were taken from each area after each dipping. Since dip is collected every three or four weeks in commercial operations the loss in weight during the first four weeks is the significant figure in the evaporation tests. Forty-eight evaporation samples were used. Of these 14 showed no loss at all in weight for the first four weeks; the remaining 34 samples showed losses ranging from 1.5 to 10.5 per cent. The average loss in weight for all the samples was 3 per cent. The gum as exposed contained turpentine, rosin, water, and chips. The loss by evaporation was of course made up of turpentine and water. The average loss of turpentine by evaporation from the cups in the Arizona experiments was therefore less than 3 per cent. The average loss in six similar evaporation tests in California was 2.5 per cent. No evaporation figures are available for southeastern operations.

COMPARISON OF YIELDS FROM NORTH AND SOUTH FACES—ARIZONA.

The total yield for 50 cups on the north side of 50 trees from the first dipping on June 3 to the last dipping on November 3 was 242.6 pounds and for 50 cups on the south side of the same trees 266.2 pounds. These weights show a 9 per cent greater flow on the south side of the trees than on the north. Figure 10 shows the average yield per week for both the north and south faces of each of the 50 trees, arranged in order of the yields from the south faces. Twenty-seven south cups yielded more than the corresponding north cups, while 17 north cups showed an excess over the corresponding south cups. The remaining six trees had about the same flow for both cups. The diagram shows the tendency of faces on the same tree to give the same yields. Trees having an exceptionally good or exceptionally poor flow generally show it in both faces.

EFFECT OF TEMPERATURE ON WEEKLY YIELD OF GUM.

Figure 11 shows the average flow per cup for 50 trees for each week and the corresponding average temperature. With but few excep-

tions the flow increased or decreased with increase or decrease of temperature. The effect of cool weather in checking the flow is especially marked toward the end of the season.

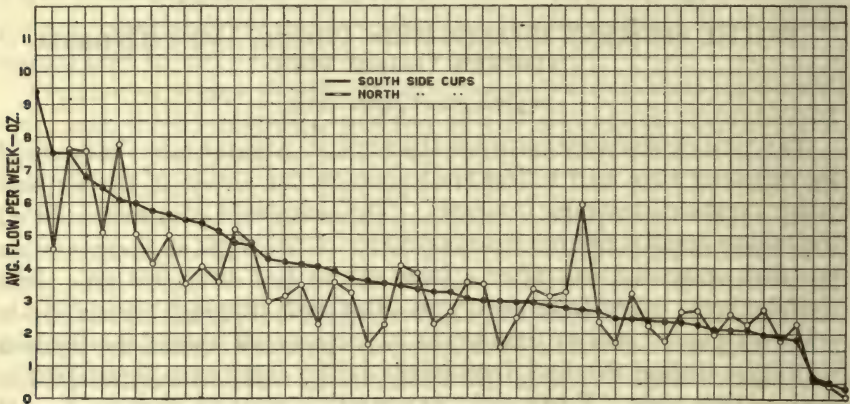


FIG. 10.—Comparison of flow from north and south faces of each of fifty trees.

RATE OF FLOW DURING WEEK.

Data on the variation in rate of flow were secured by weighing the north and south cups on 10 trees on the third day after each chipping throughout the season. The sum of the weights for each tree was compared with the weight of the total flow for the season from the

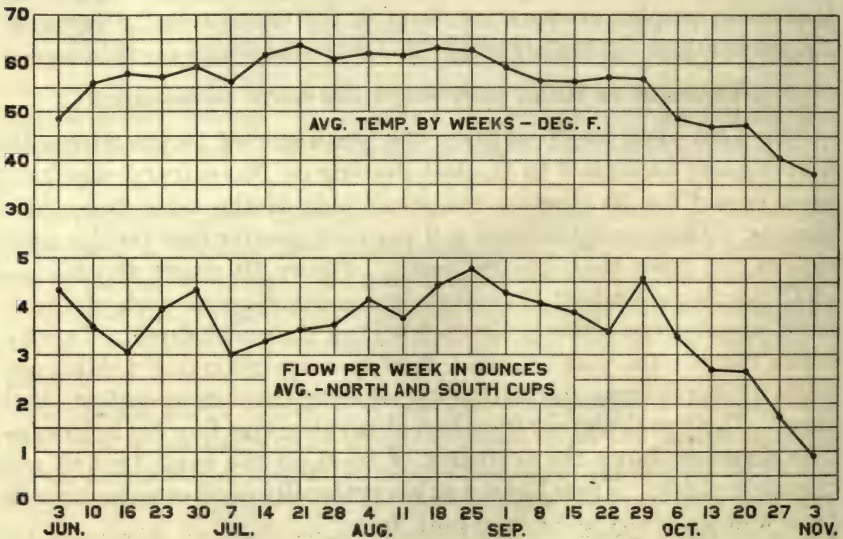


FIG. 11.—Relation between temperature and rate of flow of gum.

same trees. An average of the results shows that 73 per cent of the weekly flow occurred in the first three days. This ratio varied from 65 per cent to 78.6 per cent in the 10 test trees.

EFFECT OF VARYING FREQUENCY OF CHIPPING ON YIELD—CALIFORNIA.

Table 20 shows the yields obtained from three similar sets of 50 faces, each chipped at 3, 5, and 7 day intervals for the same period.

TABLE 20.—*Yields obtained by chipping at different intervals—California (50 faces in each set).*

3-day intervals.		5-day intervals.		7-day intervals.	
Date of chipping.	Weight of dip.	Date of chipping.	Weight of dip.	Date of chipping.	Weight of dip.
1912.	<i>Pounds.</i>	1912.	<i>Pounds.</i>	1912.	<i>Pounds.</i>
June 11....	46	June 14....	35½	June 13....	25
June 23....	49	June 29....	47	June 27....	31
July 5....	66	July 14....	49½	July 11....	31
July 17....	69	July 29....	57	July 25....	40
July 29....	81	Aug. 8....	45	Aug. 8....	45½
Aug. 10....	81				
Total ..	392		234		172½

Over twice as much gum was obtained by chipping at 3-day intervals as at 7-day intervals.

SUGGESTIONS FOR SPECIFICATIONS.

HANGING CUPS

1. The distance from the ground to the apex of the first streak shall not exceed 10 inches.

2. No "blazes" or similar scarification of the tree shall extend below the gutter or apron. The surface prepared for the placement of the gutters or aprons shall not extend beyond the ends of the same.

3. The peak of the first streak shall not be more than 2 inches above the gutter or apron.

4. The "streak" or gash made for the placement of the gutters or aprons shall not penetrate the wood of the tree more than one-half inch, the measurement to be taken from the dividing line between the wood and bark.

5. The gutters or aprons must be so attached that no "gum" flows between the tree and the gutters or aprons, or flows over the edge of same so as to fall without the cup or other receptacle.

6. For the first three years the cups and gutters shall be raised each spring to the top of the face worked the previous season.

7. A bar or strip of live bark not less than 4 inches wide in the narrowest place shall be left between the faces.

CHIPPING.

1. No tree under 10 inches in diameter shall be tapped. Minimum diameter of tree to carry one face, 10 inches; minimum diameter of tree to carry two faces, 16 inches; no tree shall carry more than two faces.

2. The face on trees from 10 to 16 inches in diameter shall not exceed 12 inches in width, and the faces on trees above 16 inches in diameter shall not exceed 14 inches in width.

3. The height of the face shall not be increased by more than 16 inches each year the tree is tapped.

4. Each streak shall not exceed a width of one-half inch or a depth of one-half inch, the depth being measured from the dividing line between the wood and the bark.

5. Before the chipping season opens the rough outer bark shall be scraped off over the entire surface to be chipped for each season, care being taken not to penetrate the living bark.

6. During the winter a space of at least $2\frac{1}{2}$ feet shall be raked free of débris about each tapped tree.

PACKING NAVAL STORES.

Buyers and exporters frequently complain that turpentine and rosin barrels reach them in poor condition, unfit for further shipment. In order to improve the standards of naval stores packages, the Savannah Board of Trade in 1911 issued letters of instruction to naval stores operators as follows:

Turpentine barrels.—All barrels, whether new or secondhand, should be kept absolutely protected from the elements, and not allowed to remain subject to rain and sunshine at way stations and river landings. Glue will not take on damp staves. Every barrel should be glued twice before being filled. Use only the best quality of glue, as it is the cheapest in the end. Before gluing, see that your pot is absolutely clean. Put into this 20 pounds of good glue and 5 gallons of water, and allow same to soak overnight. On the following morning apply sufficient heat to melt up to a temperature not exceeding 160° F. Under no condition whatever must glue be allowed to boil, as this causes decomposition to set in, which causes the bad smell usually noticed around glue sheds, and renders it utterly worthless. This amount of prepared glue will be sufficient for 20 barrels. After gluing, barrels should be taken off the trough and stood on the head for about one-half hour, after which time they should be reversed, so that the surplus glue will run down equally on both heads. The barrels should then be well and thoroughly driven, and after standing for 24 hours should be given a second coat of glue, using the exact formula as before. They are then ready to be filled in 48 hours, and if treated in this way there should be no turning except for broken staves.

Rosin.—Rule No. 9 of the Savannah Board of Trade says in part: "Rosin barrels to be in merchantable order must have two good heads, not exceeding $1\frac{1}{2}$ inches in thickness, staves not to exceed 1 inch in thickness; the top well lined." Too much stress, therefore, can not be placed on the absolute necessity of carrying out this rule to the very letter, especially regarding the thickness of staves and heading, for rule No. 10 specifically instructs the inspector to make a proper deduction in weight on all rosin when the staves and headings are more than the prescribed thickness in rule No. 9. In such cases, therefore, the operator will lose, as in addition to having the deductions made, for which he receives nothing, he must pay the full amount of freight to the railroad. Operators must see that every barrel is well coopered before shipment; see that all four hoops are nailed on the barrels, and the heads cut to fit close, and a good lining hoop as prescribed by rule No. 9 is in place. Staves must be properly equalized. Staves should be 40 inches long, and barrels built on a 22-inch stress hoop, which gives a well-shaped and easily handled barrel.

EFFECT OF TEMPERATURE ON VOLUME OF TURPENTINE.

Operators often complain that the factor's gauge of their barrels is 1 or 2 gallons less than their own. When the barrel has not leaked, the difference is usually due to the temperature at which the turpentine was placed in the barrel. Very frequently the turpentine is at a temperature of from 50° to 60° C. (from 122° to 140° F.) when the barrels are filled, and later cools to, say, 25° C. The mean coefficient of expansion of turpentine between 10° and 40° C. is 0.000817 per degree.¹ By rough calculation, assuming a specific gravity of 0.8500 at 50° C., if the original 50 gallons have cooled 25 degrees, the loss of volume by contraction will be about 1.2 gallons.²

When the condenser is incapable of cooling the distillate to ordinary temperatures, the barrels after being filled with the warm turpentine should be loosely plugged and allowed to stand several hours, or until their contents have cooled to the temperature of the surrounding atmosphere. The barrels may then be filled up to the required volume.

COST ESTIMATES ON A 20-CROP TURPENTINE OPERATION.

The cost figures which follow do not apply to any single locality; they have been derived from several sources, and are believed to cover a fair range.

The yields³ have been calculated from the data contained in Forest Service Bulletin 90. The dip is assumed to contain 15 per cent water and trash and 18.5 per cent turpentine, and the scrape 10 per cent trash and 11 per cent turpentine; while 1 gallon of turpentine is assumed to weigh 7.25 pounds.

¹ Technologic Paper No. 9, Bureau of Standards, 1912.

² The following formula will give the expansion or contraction of turpentine due to temperature changes

$$V' = \frac{GV}{G \pm (T' - T) .000817}$$

where G and V are the specific gravity and volume at the temperature T, and V' is the volume at the lower temperature T'.

³ YIELDS PER CROP, CALCULATED FROM FOREST SERVICE BULLETIN 90.

First year—31 chippings:

83,495 lbs. dip, yielding 42.8 bbls. turpentine and 199.0 bbls. rosin.

12,560 lbs. scrape, yielding 3.8 bbls. turpentine and 35.4 bbls. rosin.

Total yield..... 46.6 bbls. turpentine and 234.4 bbls. rosin.

Second year—30 chippings:

74,791 lbs. dip, yielding 38.2 bbls. turpentine and 178.0 bbls. rosin.

14,818 lbs. scrape, yielding 4.5 bbls. turpentine and 41.8 bbls. rosin.

Total yield..... 42.7 bbls. turpentine and 219.8 bbls. rosin.

Third year—29 chippings:

57,324 lbs. dip, yielding 29.3 bbls. turpentine and 136.1 bbls. rosin.

13,399 lbs. scrape, yielding 3.6 bbls. turpentine and 38.3 bbls. rosin.

Total yield..... 32.9 bbls. turpentine and 174.4 bbls. rosin.

Fourth year—30 chippings:

45,100 lbs. dip, yielding 23.0 bbls. turpentine and 107.1 bbls. rosin.

19,908 lbs. scrape, yielding 6.0 bbls. turpentine and 56.2 bbls. rosin.

Total yield..... 29.0 bbls. turpentine and 163.3 bbls. rosin.

COST OF EQUIPMENT FOR A 20-CROP OPERATION.

1 25-barrel still, with shed and fixtures complete	\$1, 200. 00 to \$1, 250. 00
20 shanties, at \$50 to \$75 each.....	1, 000. 00 to 1, 500. 00
2 houses, at \$225 to \$300 each.....	450. 00 to 600. 00
1 commissary.....	200. 00 to 225. 00
Sheds and stables.....	175. 00 to 200. 00
Tools.....	100. 00 to 125. 00
170 to 200 dip barrels, at \$2.50 each.....	425. 00 to 500. 00
8 mules, at \$200 to \$250 each.....	1, 600. 00 to 2, 000. 00
4 wagons and harness, at \$75 to \$80.....	300. 00 to 320. 00
2 horses and saddles for woodsmen, \$150 to \$175.....	300. 00 to 350. 00
1 buggy and harness.....	75. 00 to 80. 00
Cups and gutters for 20 crops, at \$45 to \$55 per thousand....	9, 450. 00 to 11, 550. 00
	<u>15, 275. 00 to 18, 700. 00</u>

COST OF OPERATION AND MAINTENANCE OF ONE CROP FOR ONE SEASON OF 30 WEEKS.

Hanging cups.....	\$100. 00 to \$125. 00
Raking and burning.....	25. 00 to 28. 00
Chipping.....	277. 00 to 300. 00
Dipping.....	120. 00 to 150. 00
Scraping.....	25. 00 to 35. 00
Hauling dip and scrape to still.....	80. 00 to 110. 00
Distilling.....	45. 00 to 50. 00
Salary of woods rider.....	70. 00 to 75. 00
Salary of manager.....	50. 00 to 100. 00
Recruiting.....	10. 00 to 30. 00
Taxes and still license.....	15. 00 to 20. 00
Cotton batting, straining wire, oil, tools, etc.....	8. 00 to 10. 00
Depreciation on cups and gutters ¹	80. 00 to 95. 00
Depreciation on dip barrels.....	5. 00 to 6. 00
Depreciation on other equipment, at 10 per cent.....	27. 00 to 33. 00
Interest on total investment, at 8 per cent.....	60. 00 to 75. 00
Lease on timber, at \$25 to \$33 per 1,000 cups.....	262. 50 to 346. 50
	<u>1, 259. 50 to 1, 588. 50</u>

COST OF MARKETING ONE CROP.

Barrels and cooperage.....	\$175. 00 to \$200. 00
Haul to shipping point.....	10. 00 to 100. 00
Selling commission, 2½ per cent.....	30. 00 to 35. 00
Inspection.....	8. 00 to 10. 00
Storage.....	5. 00 to 10. 00
Insurance, ½ per cent.....	6. 00 to 7. 00
	<u>234. 00 to 362. 00</u>
Cost of operation and maintenance of one crop.....	1, 259. 50 to 1, 588. 50
Cost of marketing one crop.....	234. 00 to 362. 00
	<u>1, 493. 50 1, 950. 50</u>
Total cost for 1 crop.....	
Cost for 20 crops.....	29, 870. 00 to 39, 010. 00

¹ The life of the cups and gutters is about 6 years.

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- 395731. BEHRENS, E. A. Bleaching and refining resins and other substances. January 8, 1889.
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FARMERS' BULLETIN No. 252.

MAPLE SUGAR AND SIRUP.

BY

WILLIAM F. HUBBARD.



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1906.

LETTER OF TRANSMITTAL

U. S. DEPARTMENT OF AGRICULTURE,
FOREST SERVICE,
Washington, D. C., March 17, 1906.

SIR: I have the honor to submit herewith a paper on Maple Sugar and Sirup, by William F. Hubbard, based upon Bulletin 59 of the Forest Service, The Maple Sugar Industry, and to recommend the publication of the same as a Farmers' Bulletin.

Very respectfully,

GIFFORD PINCHOT,
Forester.

HON. JAMES WILSON,
Secretary of Agriculture.

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MAPLE SUGAR AND SIRUP.

HISTORY OF THE MAPLE-SUGAR INDUSTRY.

The art of making maple sugar was first learned by the early settlers from the Indians.

For a hundred years or more the methods of production remained without material change save the substitution of iron or copper kettles for vessels of clay or bark, and the use of better utensils.^a The sugar was made merely for home use; cane sugar was a luxury and often unobtainable by the pioneer farmer at any cost. The trees were tapped in the Indian way and the sap was carried in buckets from the wooden troughs that stood under the spouts to the boiling kettle, or was temporarily stored in large troughs made by hollowing out logs.

The boiling was generally done in the open woods, with no protection from sun, rain, snow, and miscellaneous impurities. Green or dead and down timber, cut as fast as it was needed, generally supplied the fuel. After the sap had been boiled down to the consistency of thin sirup it was stored until enough was on hand for "sugaring off."

The crude product of early days was dark in color and quite variable in quality, while in quantity it was wholly inadequate for a large trade. In the course of time, however, the industry assumed commercial importance in the Northern States, with Vermont, New York, and, subsequently, Ohio leading in production. About the middle of the last century a rapid improvement in the methods and machinery employed took place.

PRESENT STATUS OF THE INDUSTRY.

Maple sugar, at first an article of food, has now been almost displaced for this purpose by the cheaper and unflavored cane product. Yet the demand for maple sugar and sirup for special purposes, owing to their peculiar flavor, not only keeps the industry alive but calls for a continually increasing supply. Production has not kept pace with this growth in demand, but has been more or less stationary for

^a The Indians boiled the sap down by repeatedly dropping hot stones into it.

twenty years. The explanation lies in the fact that, at the very lowest estimate, seven-eighths of the product sold to-day is a spurious article, which is only in part maple sugar or is manufactured entirely from foreign materials. The wholesale dealer, or "mixer," uses the maple sugar to flavor a body of glucose or cane sugar six or ten times as great, making a product which is marketed as "pure maple sugar."

But there has always been a certain amount of trade in pure maple sugar and sirup. A part of the city and town people come from the country, where they have known the genuine article, and they have generally been able to supply their wants by dealing directly with the producers. The progressive and well-to-do sugar maker has worked in this field. Such sugar makers are unwilling to sell their high-grade goods to the mixers at a low price, but make every effort to reach a steady market of regular customers. In the effort to make such a market more general several maple-sugar makers' associations have come into existence, which have done much to stimulate improved methods, as well as to build up a legitimate trade. But there is only a very small part of the business which is not in the hands of the mixers.

Quantity and value of maple products made on farms in the census year 1900.

[From United States Census Report.]

State.	Farms re- port- ing.	Sugar.		Sirup.		Total value.
		Quantity.	Value.	Quantity.	Value.	
United States.....	62, 718	<i>Pounds.</i> 11, 928, 770	\$1, 074, 260	<i>Gallons.</i> 2, 056, 611	\$1, 562, 451	\$2, 636, 711
New England States.....	13, 142	5, 425, 160	530, 961	246, 652	216, 510	747, 471
Maine.....	641	5, 500	643	16, 024	15, 277	15, 920
New Hampshire.....	1, 631	441, 870	44, 312	41, 588	38, 314	82, 626
Vermont.....	9, 811	4, 779, 870	464, 132	160, 918	134, 821	598, 953
Massachusetts.....	1, 000	192, 990	21, 124	27, 174	27, 112	48, 236
Connecticut.....	59	4, 930	750	948	986	1, 736
Middle Atlantic States.....	28, 650	5, 478, 100	457, 744	595, 832	466, 013	923, 757
New York.....	20, 567	3, 623, 540	307, 184	413, 159	323, 996	631, 180
Pennsylvania.....	6, 154	1, 429, 540	115, 910	160, 297	123, 863	239, 773
Maryland.....	209	264, 160	20, 561	5, 825	3, 622	24, 183
Virginia.....	213	19, 310	1, 816	1, 677	1, 534	3, 350
West Virginia.....	1, 507	141, 550	12, 273	14, 874	12, 998	25, 271
Central States.....	20, 901	1, 024, 330	85, 438	1, 213, 827	879, 638	965, 076
Ohio.....	12, 067	613, 990	48, 736	923, 519	616, 490	665, 226
Indiana.....	4, 467	51, 900	4, 372	179, 576	161, 935	166, 307
Illinois.....	248	4, 090	478	9, 357	9, 363	9, 841
Kentucky.....	93	2, 340	291	2, 367	2, 450	2, 741
Tennessee.....	64	1, 160	167	171	169	336
Missouri.....	296	12, 055	1, 288	5, 474	5, 271	6, 559
Iowa.....	83	2, 320	280	2, 662	2, 640	2, 920
Michigan.....	3, 168	302, 715	26, 693	82, 997	73, 903	100, 596
Wisconsin.....	268	4, 180	400	6, 625	6, 478	6, 878
Minnesota.....	147	29, 580	2, 733	1, 079	939	3, 672
Southern States.....	21	1, 180	117	129	117	234
North Carolina.....	21	1, 180	117	129	117	234

PAST AND PRESENT OUTPUT OF SUGAR AND SIRUP.

The history of the maple-sugar industry of the country may be read in the census figures. In the census of 1860 the total production of maple sugar and sirup reached its height. It fell heavily in 1870, rose again to large proportions in 1880, remained stationary in 1890, and then suddenly fell almost 50 per cent in 1900, when the total amount produced was nearly a third less than in 1850.

Apart from the actual decrease in product, there has been a great reduction in the area over which the maple is tapped. This decrease was 34 per cent between 1880 and 1890. The decided falling off in the distribution of the industry has taken place to a much greater extent in the area of occasional production than in that part of the country where maple-sugar making has commercial features. The tendency for sugar production to concentrate in the region of best climatic and soil condition indicates that sugar making for home use is giving way to a market production.

In Vermont the production in 1899 was especially small, not only because of the poor season, but on account of a serious attack on the groves by the "maple worm." (See p. 12.) Since that time the groves have recuperated, and Vermont is in a position to turn out as large a crop as at any time in her history.

In the Southern Appalachians and Kentucky the decrease seems permanent, and undoubtedly comes from the cheapening of cane sugar in the mountain districts. Maple sugar and sirup in those regions never had more than a local sale, and were merely used as substitutes for the cane product. But in western Maryland and the adjacent part of West Virginia the production has decidedly increased, showing that sugar and sirup are there being produced for the market, and that the southern mountains possess latent possibilities for the development of the maple-sugar industry.

In Indiana, Michigan, and Illinois the decrease undoubtedly comes from the cutting of the maples, which have been heavily lumbered in the last ten years. This is particularly true in Michigan and Indiana, both of which would otherwise be producing large quantities of maple sugar and sirup. Another cause of the decrease is the competition of adulterated products from the large towns of this section.

The table on page 6 gives the 1900 census figures of maple sugar and sirup production. The decrease for the year, both in quantity of sugar and in sugar-producing area, comes from a number of causes, some of which are permanent, but some only temporary. In the New England States, New York, and to a less extent in Ohio, the sugar season of 1899 (that reported in the census of 1900) was

very poor. For this reason the figures do not fairly indicate the present condition of the industry.

It is plain that for climatic reasons the maple-sugar industry will always center in the North, but there is nothing to prevent most of Pennsylvania and West Virginia, western Maryland, all of Indiana, and parts of Kentucky, eastern Tennessee, and western North Carolina from being included within the area of greatest production. Outside of these limits progressive farmers in favorable situations may profitably cultivate the sugar maple, but a general extension of the industry is barred by natural conditions unfavorable to the production of sap in paying quantity.

SUGAR "MIXERS" AND FABRICATORS AND THEIR WORK.

If complete figures were available to show the amount of so-called maple sugar and sirup turned out by large houses in the eastern and middle western cities, they would offer a startling contrast to the census report of the amount of maple production on the farms. (See pp. 6 and 7.) Every maple-sugar dealer in a producing section tells of the large orders for coarse sugar which come from the cities, and of the many carloads which are shipped there every year. A large amount of such sugar is also imported from Canada. It is asserted by those who have studied the trade that not more than one-eighth of the maple sugar and sirup which reaches the market is a pure product.

In the present state of our knowledge, it is difficult to determine in any particular case whether or not other sugars have been added to maple sugar.^a There are of course some differences between the maple sugar of commerce and other sugars, which serve as indications for the analyst. The consumer who is familiar with genuine maple sugar and sirup can protect himself to some extent by tasting a sample before buying. Under present conditions, however, the best guaranty of purity is the word of an honest producer or dealer.

One of the most common adulterants used is glucose. Common cane sugar is also used. A flavor like that of maple is said to be imparted to sirup by mixing with it an extract of hickory bark. An Indiana man a few years ago took out a patent to protect him in the use of such an extract from the wood and bark of hickory trees for flavoring sirup.

In spite of the well-known fact that the demand for maple sugar and sirup has increased and is increasing enormously every year, the price of both sugar and sirup has steadily declined—from 13 cents a pound for sugar and \$1.25 a gallon for sirup in 1882 to 11

^a See United States Department of Agriculture, Bureau of Chemistry, Circular 23, Method for Examination of Maple Products, by J. Hortvet. Also Jour. Amer. Chem. Soc., 1904, 26: 1523.

cents a pound for sugar and 91½ cents a gallon for sirup in 1903. Meanwhile the census returns show that the actual production of the groves has not increased since 1860. No other statement could show more eloquently the position of the trade or make clearer the fact that the grower has not profited by the development of maple sirup into an article of luxury. The cheaper the price of cane sugar the easier it is to adulterate the maple product.

By 1875 cane sugar had become cheap enough to undersell the maple, which it has now supplanted everywhere. Since 1885 maple sugar has been an article of luxury only. In this capacity its prospects are many times greater than under the old conditions; yet the grower is likely to have little share in the benefits to be reaped from this broadened field.

NECESSITY FOR SUGAR MAKERS' ORGANIZATIONS.

With a steadily growing demand for maple sirup, which to-day is almost entirely supplied by the mixer, the producers of pure sirup can hope to control the trade only through organization. The difference between the pure and the adulterated product is so marked that there would be little question as to choice, were the genuine article known to the popular trade. A large number of the consumers hardly know pure maple sirup when they taste it, and as so great a part of that on the market is spurious they have little chance to learn. Under such a condition the market can be gained for the pure product only by means of united action. An example of such action is the present Vermont Sugar Growers' Association.

A similar situation has been successfully met, in the case of certain other farm products, by organized cooperation of producers, sometimes aided by government action. Canadian dairy products, which formerly had little sale in Great Britain, are now in good demand in the English market. The Irish Agricultural Organization Society has gone far toward bringing about an economic regeneration of the island, and in Germany rural prosperity has been vastly increased by the same methods. In all these cases the principal purposes aimed at have been to improve the methods of production and to furnish a guaranty of purity to consumers.

In the case of maple-sugar producers the first necessity is a market for high-grade, unadulterated sugar and sirup. This they should be able to secure without much difficulty through responsible association, which can guarantee the quality of all the product bearing its name or stamp. Where desirable, the growers of a county or region could unite in forming a stock company, thus securing capital for the

necessary equipment. In every case all sugar and sirup should be sent to a central point, where it should be graded according to quality and packed in a uniform manner. A commission charge on sales would furnish the income from which the expenses of the association or company would be met.

Under careful, conservative business management the purchase of utensils, packing cases, etc., for sale to the members at the lowest prices, might be successfully arranged, and perhaps the loan of capital, on good security, for the installation of modern equipment could be successfully added.

Annually, if not oftener, the members should meet for the transaction of business, to interchange ideas, and to listen to addresses on matters pertaining to sugar making. The publication and free distribution of the proceedings at these meetings might be made a further means of advancing the interests and improving the methods of the sugar makers.

SUGAR MAPLES.

All the maples have sweet sap, but only from a few of our native species has sugar been made in paying quantities. The first place is held by the sugar maple (*Acer saccharum*) and a variety of it—the black maple (*Acer saccharum nigrum*). The red maple (*Acer rubrum*), the silver maple (*Acer saccharinum*), and the Oregon maple (*Acer macrophyllum*) are of less importance, and the box elder (*Acer negundo*) is least important of all.

THE SUGAR MAPLE.

The sugar maple spreads over a wide area, but as a tree for the production of sugar in paying quantities its range is limited to western New England, New York, Pennsylvania, the southern Appalachians, the Ohio Valley, and the Lake States, and adjacent parts of Canada. In the Gulf States and as far north as southern Arkansas the tree is represented by a variety (*Acer saccharum floridanum*) from which no sugar is made.

The sugar maple is a stately and vigorous forest tree, capable of growing in dense stands. It bears a plentiful crop of seeds, which in most localities ripen in the early fall. These seeds germinate readily, and under favorable circumstances the entire forest floor is heavily carpeted with seedlings, the succulent, sweet foliage of which is eagerly devoured by all kinds of stock. The young seedlings are very thrifty and can stand the shade of a complete forest cover. This tolerance of shade is one of the distinguishing features of the sugar maple, and, although it is less pronounced in later years, the mature tree has one of the most persistently heavy crowns in the forest.

Seedlings, although not killed by complete shade, are kept suppressed and grow slowly; but if they have germinated in the open, or the forest above them is removed, they grow up into thickets of remarkable density. In such a condition the struggle between the young trees is so fierce that the development of even the most thrifty is seriously retarded. The species being so tolerant of shade and by nature so vigorous, no individual gives up the struggle, but does its utmost to overtop the others and gain the sunlight. As a result the stand keeps its extreme density for a long period, and each tree grows long and spindling. The forest-grown tree develops slowly on this account, and has a long, clean stem and a small crown, while the roadside maple has a short trunk and a great egg-shaped crown of dense foliage. The root system tends to be shallow, with many laterals and an undeveloped taproot. In the forest this character is more marked than in field or roadside specimens, and any sudden opening up of the stand may result in loss by windfall or by a drying out of the roots.

There is no doubt that the quantity of sap that a tree yields stands in direct relation to the size of its crown, but many sugar makers believe that trees in a forest produce more sap than those in a grove. The explanation is found in the fact that the forest floor with its covering of litter and humus contributes to the vitality of the trees more than the grass carpet of a grove. To obtain a heavy sap production, a complete crown cover and a rich deposit of humus are of vital importance. (See pp. 14, 15, 26.)

Within its wide range the sugar maple appears as a predominant tree only in the New England States, New York, southern Canada, northern and western Pennsylvania, and in parts of Ohio, Indiana, Illinois, Michigan, Wisconsin, and Minnesota. In the southern Appalachians it occurs in scattered bodies where climatic conditions are similar to those of the North, confining itself chiefly to north slopes or to the coves, on moist, well-drained, rich soils where the heat of the sun is tempered. As a rule, it associates with the beech, birches, and basswood, but also mixes with the yellow poplar, hickories, and other hardwoods, and with hemlock and some of the eastern spruces. At the heads of the coves and in the bottoms it often forms pure stands fit for commercial tapping, and its reproduction is everywhere excellent.

In its northern home it is a principal forest tree and often forms from 25 to 75 per cent of the total stand. It prefers a moist but well-drained soil, and seems to do its best on glacial drift or on rocky hillsides and benches. In the cool atmosphere of this region all aspects are equally acceptable, but it avoids or grows sparsely on ridge crests, generally leaving the ground in such situations to spruce or to beech. In the extreme northern part of New York State and

the adjacent district of Quebec the forest growth is often almost pure maple, and even considerably farther south, where beech and birch become associate trees, the sugar maple holds its own as the dominant species. In northern Pennsylvania, at an altitude of over 1,000 feet, the mixture is much the same as it is in New York. In Ohio, Indiana, and Illinois a similar condition is found; but as the hilly country disappears the maple retreats to the richer and damper soils, leaving great areas to the oaks, chestnuts, etc. This is particularly true of the southern parts of these States. By the time the western and southwestern limits of its range have been reached it has only a scattered occurrence, even in the most favorable positions. In southern Michigan the forests are similar to those in New York, but as one approaches the pine region of the North the maple confines itself to the more fertile places. The same is true of Wisconsin and Minnesota, where the sugar maple reaches its northwestern limit in the United States.

Maple trees are often seriously injured by an insect commonly called the "maple worm," concerning which information may be had by application to the Bureau of Entomology, United States Department of Agriculture.

In the present discussion the sugar maple is not considered as a lumber tree, for which a long stem free of branches is desired, but rather as a paying producer of sap. Under this aspect a silvicultural problem is presented radically different from that which ordinarily confronts the forester. (See p. 14.) The full and heavy crown with a large leaf surface must be developed in place of the long, clear stem. The sap flow must be continuous and plentiful. The best sap flow is where the transition from winter to spring is slow, where the days are warm and sunny and the nights frosty. These conditions do not occur throughout the entire range of the species. A locality wherein the ground thaws quickly and which has no great variation of temperature between day and night is not suitable for sap production. The "season" must be long enough, also, to insure sap in merchantable quantities. Such conditions are characteristic only in the Northern States, and as sugar making goes farther south it can be profitable only at altitudes which reproduce the climatic conditions of the North.

THE BLACK MAPLE.

The black maple is generally considered superior to all others as a producer of sap. How far this is true is uncertain. In its general silvical characteristics it is similar to the sugar maple, save in the fact that it seems to prefer lower land, such as the banks of streams and rich alluvial river bottoms. It is found in Vermont on the shores of Lake Champlain, and ranges southward, west of the Alleghenies, from Minnesota to Arkansas and eastern Kansas.

THE RED MAPLE.

The red maple has the widest range of all its family in America. The natural home of this tree is along the borders of streams and on low, swampy ground. In the North it often forms a pure growth in such places, but it is along the Ohio and the Mississippi and their tributaries that it reaches its greatest perfection. Like the sugar maple it is tolerant of shade, and seedlings sprout plentifully from the heavy crops of seeds, which ripen in the late spring or early summer. As a swamp tree it associates in the Southern and Middle States with the sweet magnolia and loblolly bay, the bald cypress, various oaks, and the red, black, and cotton gums. It does well, also, on less moist lands. It is generally of vigorous growth, but the grown trees are inclined to unsoundness at the butt. As a sugar-producing tree it enters into consideration in the Middle and Western States only where the sugar maple is not plentiful. It has an abundant flow of sap, from which good sugar has been made, and the general opinion that the early starting buds cause "buddy" or discolored sap may prove quite unfounded. As a rule, red maple has been tapped in districts where the climate is unfavorable to any kind of maple-sugar making, and this fact, together with the general lack of care and skill, may account for the existing prejudice against it. Conditions being equal, it is almost certain that the sugar maple is superior to red maple in every way, but there are large districts in which no better sugar tree than the red maple is found. In such localities experiments should be made to determine its true value.

THE SILVER MAPLE.

The silver maple ranges from New Brunswick to western Florida, and west through southern Ontario and Michigan to eastern North and South Dakota, Kansas, and Indian Territory. In the North it appears in mixture with the sugar maple, but in general prefers lower altitudes and moister soils. It reaches its greatest perfection in the valleys of the Ohio and Mississippi, where it is one of the characteristic trees on the lowlands of these rivers and their tributaries. The flow of sap is plentiful and sweet, but, like that of the red maple, liable to discoloration, and the season is short and uncertain. It is, like the red maple, only to be considered as a sugar tree outside the region where the sugar maple is a dominant species.

THE OREGON MAPLE.

This is the only western species which can be considered as a producer of sugar. In localities where the season is favorable the sap is of good quality and the flow considerable. The tree is found west of the Cascades and Sierra Nevada, from the Canadian border to

southern California. It prefers rich, moist soil, and reaches its best development in the river bottoms of Washington and Oregon. The census of 1900 reports a very small production (126 gallons of sirup) from Columbia County, Wash.

SUGAR GROVES.

GENERAL CONSIDERATIONS.

The ideal sugar grove should contain that number of trees which will give a maximum yield of sap per acre; whence it follows that the formation of a grove must consider the yield per given area rather than the yield per tree. To determine the exact number of trees that should occupy an area would take many years of experiment, but directly and indirectly there has been much information collected on the subject of sap production through a study of individual trees, and from this a number of safe deductions can be made. An equal amount of sunlight being given, the sap and sugar production is proportionate to the leaf area of the tree. This statement is corroborated in a recent bulletin of the Vermont Agricultural Experiment Station,^a where it is also asserted that the sugar production of the tree depends more on the actual leaf area than on the amount of light which it receives. In other words, if a small-crowned mature tree be set free to light on all sides, the sap production will be stimulated only to a very slight extent. From this it follows that the number of trees per acre must be consistent with the greatest possible crown development of each tree in the grove. At the same time it is not to be forgotten that the maple is inherently a forest species. The large crown of foliage has an extensive leaf area for evaporation, and demands a protected soil which can keep it well supplied with water. Such soil is best found in the forest, where the ground is kept heavily matted with leaves and humus, so that the sun and drying winds will have little access to it, and a comparatively uniform degree of moisture and coolness may be maintained under all conditions. Commercial sugar making is confined to a small part of the botanical distribution of the sugar maple, because of a peculiar climatic requirement. It is the gradual northern spring, with the slow yielding of the frost by the ground, which makes the sap flow long and continuously enough to give a paying production of sugar. A sudden thaw affects both the quality and the duration of the sap flow. On this account it is always desirable to maintain forest conditions in a sugar grove, for if the ground has a heavy carpet of leaves and humus, it will be less sensitive to changes in temperature.

^a Bul. No. 103, Vermont Agricultural Experiment Station, Dec., 1903, pp. 117, 118.

Altitude is one of the most important factors in determining the necessary density of the sugar grove. High up in the mountains, where the summer is moist and the spring long, and in the North, the necessity of an unbroken cover is not so great as where the summer is hotter and the spring less gradual in its transition from winter to warm weather. In mountainous regions the forest can be more open, and in every large grove a section on a southern exposure will insure an early sugar season. In lower altitudes the close grove of full-crowned trees will have an advantage over a scattered stand of field trees exposed to the effects of a variable spring. It should not be forgotten, however, that trees which have developed from their youth in very open groves have stronger root systems than forest trees, and that they draw their water supply from the moist subsoil (see p. 11); but such groves have a relatively limited production per acre, and, while serviceable for a small home production, would cover too large an area to be profitable for a large undertaking.

The model grove should satisfy the following requirements as far as possible:

(1) It should contain the greatest number of trees per acre consistent with fully developed crowns.

(2) The forest cover should be unbroken, so that in summer little sunlight falls upon the ground.

(3) There should be a complete litter of humus and leaves, to the exclusion of grass and light-demanding weeds.^a

(4) Young trees should be kept in reserve to take the place of those that fail, and to fill other openings in the cover.

(5) No grazing should be allowed in the grove, except in special cases where the cover is perfect and no reproduction is needed. Cattle not only keep back all reproduction, but also do harm by trampling and breaking the ground, so that it dries out.

(6) The grove should be made accessible by a system of roadways to facilitate the collecting of sap. If the network is complete no difficulty will be found with the underbrush, which hinders sap gathering little in the early spring when the woods are devoid of foliage.

The first three points vary in importance with the latitude and altitude of the grove, but they are always worthy of consideration.

In discussing the methods required to bring about these results, the several common types of sugar groves will be described. Logically it would be proper to begin with the treatment of a sapling thicket and continue through each stage to the mature grove, but as the earlier stages of growth are the most complicated to deal with, the order of consideration will be reversed.

^a French and German experiments have demonstrated that a heavy growth of grass dries the soil, and interferes with the entrance of water even during a heavy rain.

THE IMPROVEMENT OF A MATURE DENSE GROVE.

A large number of groves are merely parts of the old hardwood forest, having a preponderance of sugar maple in the mixture. These trees have their normal forest form—a long, smooth stem and compact crown. There is little to be gained in actual sap production by thinning such a stand (p. 14), as it has generally passed the period of vigorous growth and would not develop larger crowns, although the sap season may be brought on earlier by opening up the grove to sunshine. The mixture can be regulated, however, and provision made for a pure growth of maple to succeed the old forest as it passes away. The usual mixture of birch, beech, elm, basswood, and ash may be gradually removed, and the reproduction of maple thereby assured. This thinning should pay for itself in most localities, from the resulting fuel and saw timber. In making such a thinning the following precautions should be observed:

(1) When the trees to be removed occur in groups, they should not all be cut out immediately, leaving large gaps in the forest cover, since forest-grown sugar maples have a broad, shallow, root system, and are subject to windfall when suddenly exposed. The trees which crowd the best maples should be taken out first; the rest should be removed later, when the sugar trees have become more wind firm.

(2) Where several maples crowd each other and form a dense cover, those with the smallest crowns, those which are unsound, and those which show signs of bad health or decline should be removed.

(3) Young maples which show possibilities of good crown development should be cut free from interference on every side.

(4) If the grove borders on open land, it should not be thinned for a distance of at least 25 feet from its edge. This is a safeguard against damage by storms, and is particularly important in borders exposed to heavy winds.

(5) When practicable, the young growth of other species than maple should be removed.

(6) It is well to accomplish the thinning in a series of years, rather than at once and radically, thus avoiding violent changes.

(7) It is important to maintain the humus and ground moisture in every maple grove. In localities where natural forests of sugar maple are common the danger of destroying the proper soil conditions by letting in the sunlight is not great, but if a grove of this type lies where the summers are hot the cover must be broken very gradually.

THE IMPROVEMENT OF A MATURE OPEN GROVE.

In the more settled and less wooded portions of the maple sugar producing district, it is noticeable that a large proportion of the groves

are old and very often overmature. They have evidently been left on favorable situations from the original forest, and as a rule no attempt has been made to renew them or keep up their vigor since the adjoining land was first cleared. A young and thrifty set of trees is a rarity among the great number of old, open, and grass-grown groves.

As a rule these groves are on small farms, where they are used quite as much for pasture as they are for sugar making. In cases where the pasturage can not be spared, and where sugar is only a small item in the farm production, there is little to be done for their improvement. When the grazing can be spared, however, and the owner desires to increase the sugar-producing capacity of his trees, it is undoubtedly better to bring about a reproduction from the old trees than by planting a new stand.

The first step to be taken in such a process of improvement in a more or less open and grass-grown grove is the exclusion of stock.

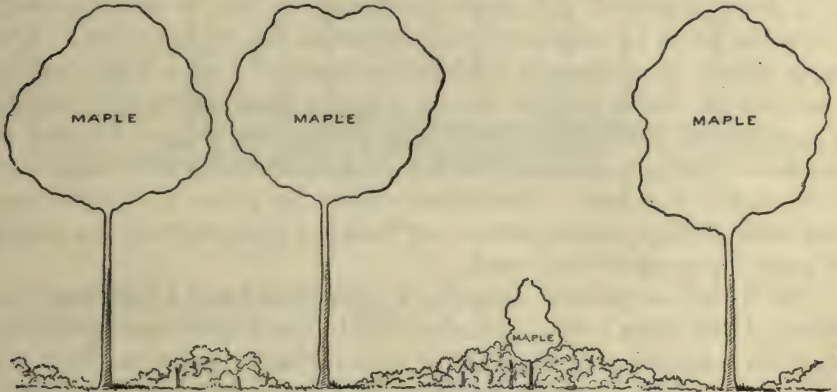


FIG. 1.—Method of improving an open grove.

After laying out proper driveways for sap gathering, the seedlings should be allowed to come up everywhere else. All unsound and dying trees should be cut out and young growth of all other species than maple removed. In a very short time the young maple seedlings will take possession of the open ground and grow vigorously where they get sufficient light. When they are 8 or 10 years old and 6 to 8 feet high, or more, the struggle for supremacy among them will begin. In each opening large enough to permit the development of a tree with a full crown, the strongest and most thrifty seedling which has a favorable position should be selected, and the heads of those within a radius of 12 feet or more about it lopped off with a corn knife. The crowns of at least two-thirds of these trees must be removed; the remaining crowns will insure a good ground protection and leaf fall until the favored tree has filled the opening. In the case of small openings the thicket should remain unthinned; the struggle

between the trees will keep them all suppressed, while they will supply the necessary ground cover. The seedlings which come up under the direct shade of the old trees will never develop to any size, unless some of the large trees are removed by age or accident. Figure 1 shows this method of treatment. Cattle may be let in the grove when it has become too tangled for convenient sap collecting, and when the young growth desired for open places has reached a height of 8 or 10 feet. They will soon open up the smaller and undesirable growth. At the same time roadways should be opened and the ground kept free of fallen limbs and trees. The tall, slender seedlings will be a small obstruction in sap gathering, but a little discomfort can be borne for the sake of the undoubted advantages obtained by a ground cover.

THE IMPROVEMENT OF A DENSE YOUNG GROVE.

In many parts of the maple-producing section a second-growth forest has come up similar in composition to the original stand. The sugar maple often forms a predominant part of such a wood, and in that case all that is needed to turn it into a sugar grove is to remove a number of interfering trees, thus giving the proper number of maples a chance to develop the full crowns necessary to a maximum yield of sap per acre. Preference should be given to the younger and more thrifty stands, where the trees are just entering the period of most vigorous development.

The difference between thinning a young stand and a fully matured grove of the same type is usually that in the former case provision must be made for growing a set of full-crowned sugar trees from the more thrifty of the young maples. In a stand from 40 to 60 years old it is easy to pick the largest and best-developed specimens and favor them for the future. Some of the directions to be observed in treating a dense young grove are the same as those given for the treatment of the mature grove.

(1) Select the sound, dominant trees which show a natural tendency to a well-branched, compact crown of large size, and remove from all sides everything which tends to crowd them. If the stand is between 40 and 60 years old, leave about 100 trees to the acre; if older, leave about 75 trees. The average healthy young maple can be freed for 10 to 12 feet on all sides of its crown without the slightest danger, except in the most exposed positions.

(2) In the choice of sugar trees the position and influence of each on its neighbors must be considered. If two dominant trees crowd each other seriously, remove the least promising.

(3) In case the beech, birch, or other species are so grouped that their removal would make a serious gap in the forest, it will be well

to let several of them stand, but they should be so treated that maple seedlings (which nearly always gain possession of the soil even under beech) will have light enough to come in under them. When these seedlings become established the beech or birch can be removed, and young maples favored after the method shown in figure 2. When practicable always cut out other seedlings than maple.

(4) Successive thinnings are better than a radical opening up of the stand, because in this way danger of windfall and drying out the soil

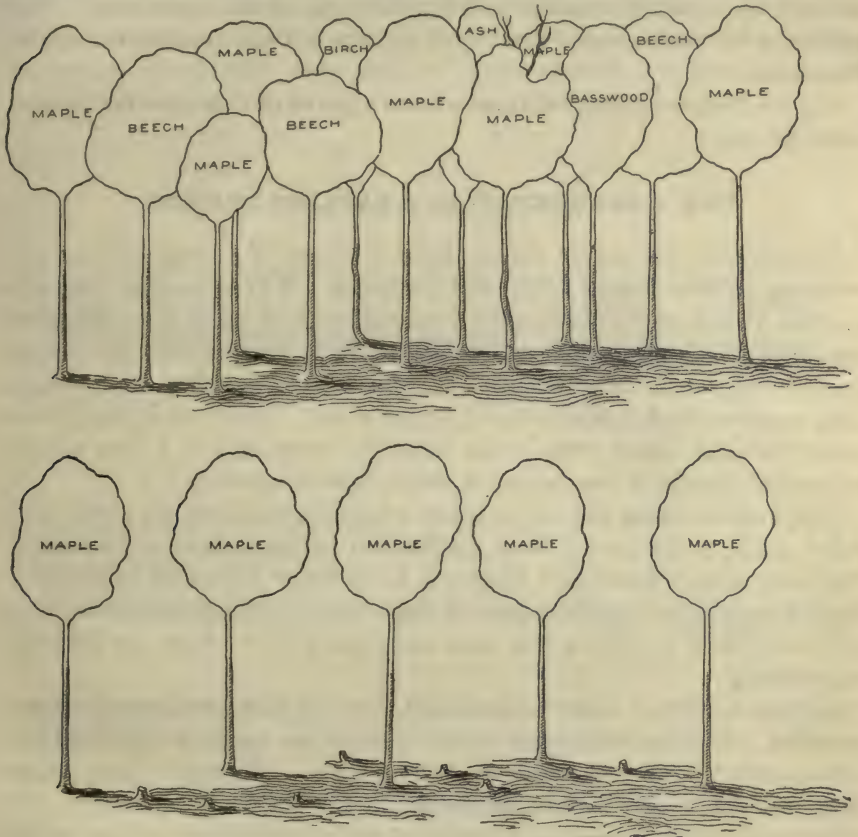


FIG. 2.—A young, close-grown grove before and after thinning.

are avoided. This method also leaves room to overcome the damage done by porcupines. These animals probably are the worst enemies of the young maple. One porcupine in a single night can strip the bark off many saplings, and to such an extent that they are permanently ruined. The sugar trees should not have more than 10 or 12 feet of free space on any side of their crowns. A thrifty maple can fill such a gap in eight or ten years, after which a final thinning may be made and the remaining weed trees removed.

(5) The edges of the grove which border upon open land should not be thinned enough to leave the stand unprotected from strong winds and sunlight. If other species are crowding the dominant maples, they should be removed; but, as a rule, the borders should remain dense and the trees should be covered to the ground with foliage.

(6) In cool situations, or in elevated regions, the thinning may be heavier than farther south or in lower lands, where more care is necessary to preserve a proper ground cover. Firewood and other timber secured by thinning should pay for the cost of the operation. The necessity for well-located roadways to take out the sap should not be forgotten.

Figure 2 shows in diagrammatic form a grove of this type before and after thinning.

THE MANAGEMENT OF A SAPLING THICKET.

Throughout the maple region dense thickets of young saplings are common in abandoned fields and pastures. Where a sugar grove is desired, it will pay to take such young growth in hand if no old trees are available in sufficient numbers. Left to themselves, the young trees usually become so densely crowded that even when 20 feet high they number from 2,000 to 3,000 to the acre. Under such conditions growth almost ceases even in the dominant trees, and at a time which, in normal stands, is the period of most vigorous growth.

The first thinning should be made when the saplings are about 6 or 8 feet high, if the owner feels justified in helping them at this time. The largest and healthiest trees, on an average about 12 feet apart, should be selected, and the tops of the others cut back with a hatchet or a corn knife in such a way that they can not overtake the favored individuals.

Figure 3 gives a conventionalized view of the treatment recommended. Cutting back trees in this manner can be done very rapidly. Three men should cut over an acre a day. Although there is no return in firewood or other material from such early thinnings, the young trees will be given a favorable start in their development at the most critical period of growth. The ground cover at the same time will be kept intact by the sprouts, until the selected trees fill out and close up the space with their crowns. When they are about 25 years old the dominant trees, which are about 12 feet apart, will begin to crowd each other, and another thinning must be made to give the best ones room. Experiments are under way to show how much time will be gained by this method in obtaining a stand fit for tapping. All general forest practice shows that the gain over the unthinned thicket should be at least 25 per cent.

If the thicket to be turned into a sugar grove contains older and larger trees than have been considered, a regular course of thinning should be instituted. The main points to keep in mind in this case are as follows:

(1) Choose the thrifty trees which show a tendency to good, symmetrical crown development, and set their crowns free on all sides to a distance of about 12 feet. See that the selected trees are sound and free from forks which may develop badly.

(2) Remove all long, spindling trees which are likely to bend over.

(3) For ground cover, leave all specimens which do not threaten the crowns of the chosen trees and which are capable of casting a small amount of shade.

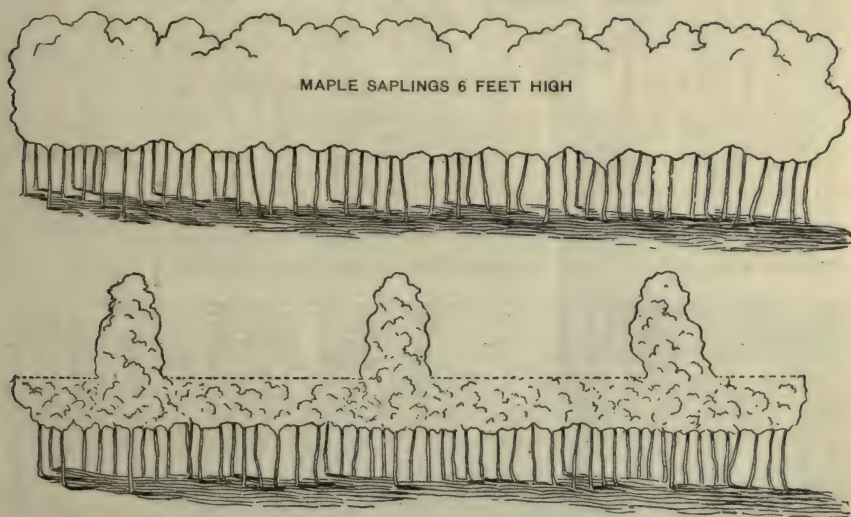


FIG. 3.—Method of improving a thicket of maple saplings.

(4) Remove all species but maple, except when they are very much suppressed. Low, broad-crowned trees of any kind will help to shade the ground.

(5) Do not disturb the borders of a dense thicket. Sun and wind must be excluded from a stand which has been suddenly opened up within, and which is unaccustomed to the new conditions.

To make this system of treatment clearer, a concrete case will be described. In the autumn of 1903 a stand of young maples in Vermont was thinned by a member of the Forest Service. The stand is situated at an altitude of about 1,200 feet, with a southeastern exposure. It came up in an abandoned meadow, which was seeded from a few old trees along a bordering wall. The dominant trees are from 30 to 40 feet in height and from 15 to 25 years old, with an average diameter for the stand, suppressed trees included, of 2 inches,

breasthigh. The entire tract is very dense, and, although the extreme difference in the age of the trees is about ten years, the difference in their size is far greater than the discrepancy in age would explain. More than half the stand is 1 inch and under in diameter, and yet many of these trees are as old as near neighbors three times as large. This results from the extreme vitality of the sugar maple, and shows the urgent necessity for thinning at an early age. Two plats, each 0.7 acre in size, were thinned, with the following results:

Thinning of a maple-sugar thicket, showing the number of trees per acre of various diameters in the original stand, the number removed, and number left.

Diameter breasthigh.	Number of trees per acre.		
	Original stand.	Removed.	Left.
<i>Inches.</i>			
0.5 to 2.....	1,517	755	762
2 to 4.....	1,042	653	389
4 to 7.....	309	126	183
Total.....	2,868	1,534	1,334

Figures 4 and 5 show the tract before and after thinning. Twelve cords to the acre of fair firewood were cut, an amount which should



FIG. 4.—Stand of maple saplings in need of thinning.

ordinarily pay for the thinning. The large number of small trees left after thinning is noticeable in the illustration, and is a point not to be overlooked. All trees that in no way interfered with the dominant

stand and had a fairly full crown were allowed to remain as cover. There is no chance of their overtaking the favored trees, and they furnish the needful shade whereby a more radical opening of the crowns in the dominant stand is permitted. The final trees of the grove are to be selected from the trees which are 4 to 7 inches in diameter, the remainder acting as a reserve in case the selected trees should meet with accident. The heaviest cutting was made in that part of the stand which ran from 2 to 4 inches in diameter, the class which interfered most with the future sugar trees. Those individuals which gave promise of becoming members of the final stand were given more room than the others. Although the cutting took away such a large



FIG. 5.—The same stand shown in fig. 4 after thinning.

proportion of the stand, it will be observed that the trees are still in close order. This will necessitate a later thinning, probably after about six years, but at present further thinning would subject the long, slender saplings to danger of overthrow and the ground to drying.

SITUATION OF A SUGAR GROVE.

The best location for a sugar grove is where the maple thrives best under natural conditions. In the Appalachian region this will be in the north coves, and in Ohio, Indiana, and adjacent States on rich, moist, gravelly soils. In the Northern States, where the maple flourishes on all exposures, the exposures to the south are generally to be

preferred, because there the sap runs earlier, and the first sirup and sugar to reach the market obtain the best prices. On northern exposures and in very dense forests the sap season begins later; but if the sugar grove is to be on a large scale, it will be well to have it include both southern and northern exposures, so that the run of sap may be continued longer and not come at once in a quantity too great to be easily cared for. In the Northern States the best sugar groves are usually on rocky slopes with soils rich in humus, at an altitude of about 1,000 feet.

PLANTING A SUGAR GROVE.

The advisability of planting a sugar grove will depend partly on the locality. The problem presented is notably different in the Middle West and in the region of commercial production in the Lake States and the Northeast. In the West maple-sugar production has steadily declined and shows no sign of a revival. The planting of sugar groves in this region is, therefore, not generally advisable.

In the region of commercial production it is usually easy to find old groves, young stands of second growth, or sapling thickets which can be made productive more quickly than a plantation of seedlings. In cases where no such beginning is possible, and a plantation has been determined upon, the following suggestions may be useful.

Avoid planting the trees too far apart. This is the mistake most commonly made. Wide spacing deprives the soil of its needful protection, reduces the yield of sap per acre, and gives a poor return for the expense of planting and for the amount of land used. Planting should always be done in early spring; and as the regions in which it is likely to be necessary are usually at low altitudes (see pp. 11, 12), it will be good policy to plant the trees close enough to insure a proper ground condition from the first. This will be best accomplished by setting the trees 6 by 6 feet apart. This gives 1,210 trees to the acre, which will not prove very expensive, as small seedlings, costing about \$2 per thousand, may be used, or they may be gathered from the woods, preferably in wet weather. When this is done, care should be taken to select thrifty specimens, not over 2 feet in height, and to plant them immediately.

When the young trees reach a height of about 10 feet and begin to crowd one another, the grove should be treated in the same manner as that recommended for the wild sapling thicket (see p. 21). This will give a maximum number of full-crowned trees to the acre, and the proper ground conditions will be maintained.

In most cases it will be well to cultivate the ground for one season, or possibly two, but the soil should acquire the forest character as soon as possible. Where that is not readily attainable, a maple grove is not likely to pay.

In some situations it may be advisable to mix with the maple a number of quick-growing trees valuable for posts or farm lumber, in order to secure early returns on the investment. The best species to use in this way can be determined only for definite localities. Advice in such cases will be willingly furnished by the Forest Service.

MAPLE SAP.

The quantity of sap produced depends not only upon the size of the tree, but also on its relative situation, and often upon what seem to be peculiarities of the individual tree, not yet explained.

SAP PRESSURE AND FLOW.

It has been recently shown that the force exerted by the sap of maple trees in the sugar-making season varies from a suction of 2 pounds per square inch at night to a pressure of 20 pounds per square inch in the day, and that it fluctuates in a general way with the rise and fall of temperature during the day and night.^a Although the phenomenon of sap flow is not yet perfectly understood, it may be asserted that the popular idea of sap rising in the spring and retreating to the roots of the tree in the autumn is a fallacy. Conclusive experiments have shown that on tapping the tree a flow of sap both from above and below comes toward the holes. There is also a very small flow from the sides of the tap holes, the sap moving freely up and down the grain of the wood, but very slowly and in small quantities across it. Since the flow varies with the season, the day, and the variation of temperature between day and night, different quantities of sap are yielded by the same grove and the same trees in different years. These circumstances make an average yield very difficult to estimate. However, it may be said that an ordinary mature and thrifty maple will produce about 12 gallons of sap, or 3 pounds of sugar, per season. This figure is not extreme, for a sugar grove has been known to average 19 gallons of sap per tree during eight consecutive seasons, which included one poor year. Some trees have been credited with enormous yields. For instance, a tree in Vermont is known to have produced 30½ pounds of cake sugar in one season, its sap being so rich that 7 quarts made 1 pound of sugar. Another maple in the same State gave 175 gallons of sap in one season.^b

There is no doubt that large-crowned trees yield the most sap, so that those trees grown in the open produce the greatest quantities. A too open grove, however, is not to be recommended, because the yield per acre is necessarily less from the smaller number of trees, and

^a Bul. 103, Vermont Agricultural Experiment Station, 1903.

^b Timothy Wheeler: Proceedings of Vermont Sugar Makers' Association, 1900.

the ground, from lack of proper cover, is more exposed to undesirable extremes of temperature during the sap season. The maple is a forest tree, and should grow under conditions that approach as nearly as possible those of the forest, but there is no reason why the grove can not be so worked that large, full-crowned trees shall occupy the ground and at the same time keep the soil sheltered and the forest cover perfect.

THE AMOUNT OF SUGAR IN MAPLE SAP.

Maple sap is a nearly colorless liquid composed of water, sugar, and various mineral substances, such as lime, potash, magnesia, and iron; it also contains some organic matter in the form of vegetable acids. The peculiar flavor of maple sugar comes, not from the sugar, but from some one or a combination of all the other substances contained in the sap.

The amount of sugar in the sap of the average sugar maple tree varies greatly, the percentage changing in each tree as the season progresses. Careful experiments have shown that the sap contains on an average about 3 per cent of sugar. The maximum is reported at 10.2 per cent, which was found in a small flow of sap from a sugar maple near the end of a season, during which the tree averaged 5.01 per cent.^a

THE MANUFACTURE OF SUGAR AND SIRUP.

IMPROVED METHODS.

The increased demand has brought about the introduction of economical methods in manufacture in sharp contrast with the primitive methods which once prevailed and which still persist where production remains on a small scale. The auger hole and the closed metal spout have been substituted for the old destructive ax cut and open wooden spout. Similarly, tin or galvanized-iron pails have gradually taken the place of the rough wooden troughs which formerly caught the sap.

At first the sap was generally carried to the fire or sugarhouse in buckets by hand or with a shoulder yoke. Occasionally a barrel and sled, drawn by an ox team or horses, were added to the gathering outfit. But as the scale of operations increased, the gathering tank was introduced and is now used in all but the smallest groves. Where the work is on a large scale, pipes are often run through the bush, as the grove is sometimes called, connecting with the sugarhouse or with large storage tanks on the roadside, while in one large Adirondack sugar grove a narrow-gauge railway is used for collecting sap.

^a Dr. H. W. Wiley: Bul. No. 5, Bureau of Chemistry, U. S. Department of Agriculture.

The change in the actual process of sugar making is absolutely revolutionary. This is chiefly due to the supplanting of the iron kettle for boiling the sap by the modern evaporator, an apparatus remarkable for the ingenuity displayed in its construction and for its adaptability to the needs of the industry. Evaporators are open pans about 6 inches deep, 40 inches wide, and from 10 to 18 feet long. They are often made with corrugated bottoms, to increase the heating surface. Partitions from side to side and open at alternate ends are placed in them at intervals of from 8 to 10 inches. (See fig. 8.) The sap, whose flow from the storage tank is carefully regulated, enters the evaporator at one end and flows slowly across the pan from side to side, around the partitions, until it reaches the far end. By that time it is reduced to the desired density.

The rate of sap flow into the evaporator is of the utmost importance. All of the latest models have automatic regulators, by which the inflow of sap increases or diminishes with the heat under the pan, and the sap is entirely cut off when the fire gets low. With such an arrangement no scorching of the sap is possible unless the supply tank becomes empty.

Improvements in the method of firing have kept pace with those in boiling. From the old, rough fire box has been evolved the modern portable arch, made of iron, with a flue running beneath the evaporator. It is lined with fire brick, and has grate bars and accurate dampers, so that the heat is more regular, while no smoke is allowed to escape. Its economy of fuel is many times greater than that of the old fire box. (Fig. 9.)

The sugarhouse has advanced with other improvements and in well-ordered groves to-day it is a neat, well-built affair, with two rooms and a woodshed. The utmost cleanliness is insisted on and maintained.

It must not be supposed that the adoption of these improved methods has been universal. They are practicable chiefly for large operations. There are still many parts of the country in which sugar is produced only in a small way, or for home use, and almost every form of sugar making, even the primitive, may yet be found. But in the great producing centers of the North Atlantic and Lake regions, which mainly supply the market, improved methods are almost universally practiced.

THE SAP SEASON.

The sap season throughout the maple-sugar belt of the United States generally begins about the middle of March and continues until the third week in April, but it varies very widely with a late or an early spring. Sugar making has begun as early as February 22 and as

late as the first week in April. The season lasts on an average about four weeks. The longest run on record included forty-three days, and the shortest eight days.

PRELIMINARY PREPARATIONS.

Before the sugar season opens, the necessary stock of dry wood for fuel should be provided. Professional sugar makers cut their wood supply six months or more before it is needed, so that it will be well seasoned. Seasoned wood is most conducive to efficient work, but, in its absence, green wood can be used. Yellow birch, in particular, makes a good fire, even when newly cut. The necessity for fuel makes thinnings and improvement cuttings in the sugar grove practicable (see pp. 16, 21). The utensils also should be made ready for use. (For list and prices see p. 36.) Spouts, pails, gathering tank, storage tank, and evaporating pans should be thoroughly cleansed by scalding them with boiling water. Absolute cleanliness is the watch-word of good sugar making.

TAPPING THE TREES AND GATHERING THE SAP.

Before tapping a tree, any loose bark which may fall into the sap should be brushed away from the trunk. For this purpose an old stiff broom or brush is useful. Select a spot on the sunny side of the tree if possible, and, avoiding defects or old scars made by tapping, bore a hole 1 inch in depth with a three-eighths or half-inch bit. The hole should be directed slightly upward to insure drainage. Trees under 12 inches in diameter at breastheight should not be tapped, and except in the case of the largest and most productive maples there should be but one spout to a tree.

The spouts should be of metal, preferably of malleable iron and heavily tinned (fig. 6, Nos. 8, 9, and 10). These are most lasting, but tin ones can be obtained at a slightly smaller cost. Wooden spouts are usually made of elder or sumach, out of which the pith has been forced with a stick, or burned with a hot iron. When these are used, a supply of them should be made before the sap season and stored in a cool place, where they will not check or warp.

Sap pails should be of tin or galvanized iron; wooden ones are less durable and harder to keep clean (fig. 6, Nos. 2 and 4). If wood is used, the pails should be enameled white on the outside, as a protection against heating by the sun. It is best to keep the pails covered, for rain, snow, or dirt will cause a deterioration of the sap and darken the color of the sugar and sirup. If sugar making is undertaken on a large scale, it is well to have the covers reversible, with one side painted red and the other white (fig. 6, No. 6). By turning the red side up when the pail is emptied the gatherer will know it does not

require his attention. When covers are used, a pail is hung to the spout by a hole in its rim. In this way the sap falls into a closed



FIG. 6.—Sugar-making utensils: 1, sugar mold; 2, 4, sap buckets; 3, gathering pail; 5, skimmer; 6, cover for sap bucket; 6a, cross section of same; 7, gathering tank; 8, 9, 10, sap spouts.

space, and the danger of evaporation and souring is greatly lessened. It is usually advisable to throw away the first drippings, for they are

apt to be mixed with dirt, or to turn sour and contaminate the later flow. The sap runs very clear at first, but as the season advances it shows a tendency to darken. The most probable explanation of this is the oxidation that takes place about the hole.

DEPTH OF TAP HOLES.

The relation of the depth of the tap hole to the amount of sap secured has been much discussed. A nearly uniform depth of about 1 inch is now used by professional sugar makers. The sap is found almost entirely in the sapwood, which in the mature maple is confined to about twenty-five or thirty annual rings, or from 2 to 3 inches of the outer wood, according to the tree. The great bulk of the sap, however, is confined to the outer ten or fifteen rings of growth. The increased cost of deep boring is not offset by the increased yield, while the injury to the tree is considerably greater. The sap also is of darker color and considered inferior by sugar makers.

The first requisite for transporting sap to the sugarhouse is a good system of roads throughout the sugar bush (p. 15). In some respects sap is as delicate a product as milk, and the method of bringing it from the tree to the storage tank must be rapid and systematic. In small groves the carrying can be done by hand, of course, or with the old shoulder yoke, but with larger operations the transportation must be effected by horses, steam power, or gravity, and must be fully organized.

If the grove be of moderate size (from 15 to 25 acres), teams may be used to haul the sap in a gathering tank (fig. 6, No. 7) on sledges or stone boats. The labor of carrying the sap by hand to the hauling tank will be in proportion to the number of roads and their proximity to the trees. The tank should be metallic, but if of wood it should be painted white on the outside, to keep the sap cooler and prevent souring. When the grove is situated on a steep hillside it will often pay to run a pipe line, with receiving funnels at regular intervals, for the conveyance of the sap to a lower storage tank or directly to the sugarhouse. The storage tank should be of tin or galvanized iron, encased with wood and covered, to keep the sap cool and to prevent it from freezing. Every practicable precaution should be taken to keep the sap in good condition and free from impurities. As it is very sensitive to changes in the weather, and is likely to sour if it becomes heated, it should be collected regularly, and as soon as possible after it has left the tree. Some sugar makers begin to gather sap as soon as there is a quart or so in each bucket, even at the expense of going over the ground twice in a single day. The gathering tank should have a strainer over the mouth, and the storage tank should be kept at an even temperature, even if it must be cooled with ice during a sudden period of heat. Often during the

season the sap runs slowly or stops altogether. Such an occurrence may be taken advantage of to wash and scald the gathering tanks, storage tank, and evaporators.

THE EQUIPMENT.

The sugarhouse is the most important adjunct to the grove, and should be planned with reference to the scale of operations under-

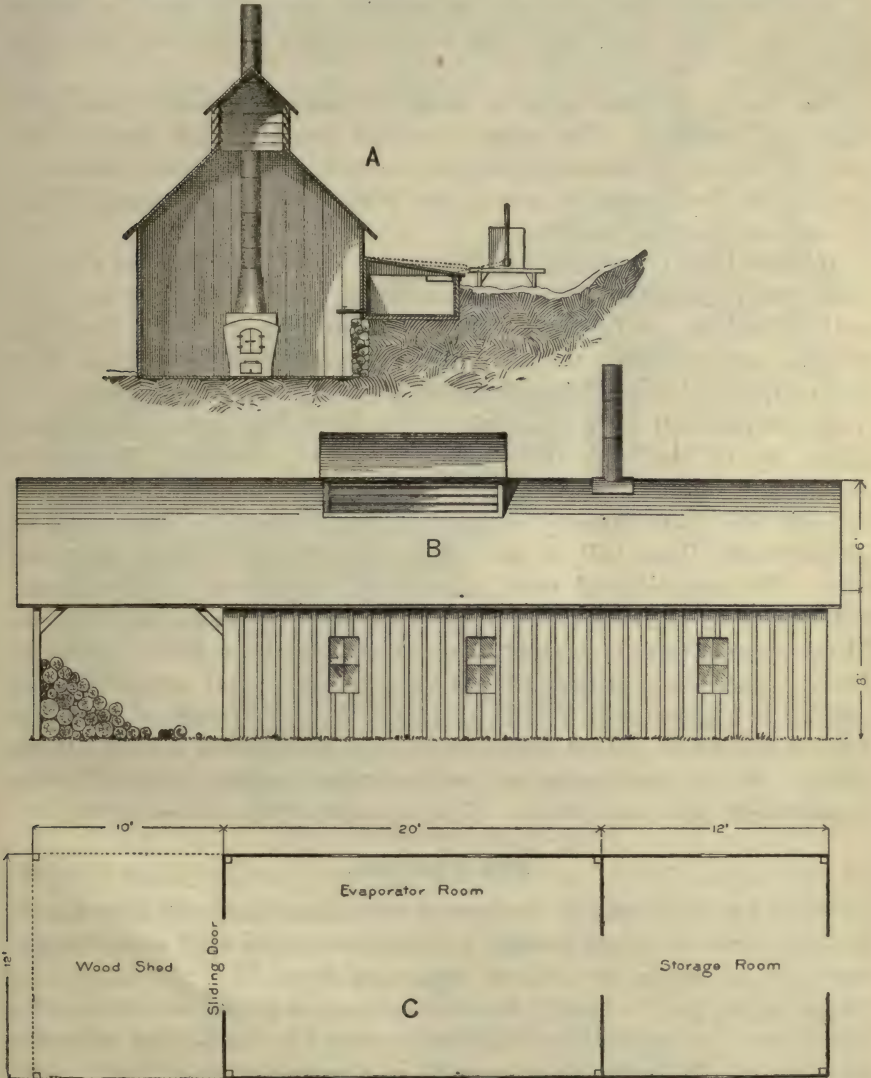


FIG. 7.—Plan of model sugarhouse: A. Sectional view, showing evaporator, storage tank, and gathering tank (on sled). B. Elevation. C. Ground plan.

taken. If only a few trees are tapped the work may be carried on in the old-fashioned way, but even in such a case it is better to

have a small, one-roomed house with a woodshed. In this room the small evaporator and sugaring-off kettle may be placed, and the work carried on satisfactorily. It is always well to put the sugarhouse on sloping ground, and, of course, in the most convenient place in the grove. If the ground rises above the house, the storage tank can be readily filled from the gathering tanks, and at the same time fed by gravity into the evaporator. If the grove be on level ground, it will generally pay to make an artificial elevation upon which to place the storage tank, with room for the sledges to pass and discharge sap into it. (See fig. 7.)

The house, whether large or small, is best when built thoroughly tight and shingled. The space overhead should be left open to the roof, in which a ventilator should be built over the evaporator to permit the free escape of steam, but with the slats so arranged that no rain or snow can enter.

If 1,500 trees or more are to be tapped, it will pay to have a house with two compartments and a woodshed, something after the general plan shown in figure 7. The evaporator room should form the middle compartment; the room for sugaring-off should adjoin it at one end, and the woodshed at the other. For the evaporating room 12 by 20 feet will be a convenient size, with the sugaring-off room of 12 by 12 feet and the woodshed slightly smaller. The latter should be open in front, with a wide sliding door opening into the evaporating room directly in front of the fire arch. With such an arrangement there will be no dust or dirt blown in from the wood pile. The sugaring-off room should be lined with stout shelves, which will be convenient in handling and storing the maple products. If the storage tank is heavily cased in wood and not likely to freeze, its best position is outside of the sugarhouse on that side where the ground is highest, but if it is in danger from extremes of temperature, it should be placed within the house, even at the cost of a little room. Such a sugarhouse can be kept clean and airy, and will prove comfortable and convenient. The cost should not exceed \$125.

THE PROCESS.

What has been said of the perfect appointment of the sugarhouse, be it on ever so small a scale, is true also of the appliances, implements, and actual process of sugar making. The best models of evaporators can be bought in all sizes, and a good one will pay for itself, even in the smallest undertaking (fig. 8). In boiling down sap to sirup, the following points are to be observed:

The best modern evaporators are made so that the sap feeds automatically into the pans, running fast or slow according to the heat under the pan. Although the pans are about 6 inches deep, the sap in

them should be kept very shallow, the best results being obtained from a depth of $1\frac{1}{2}$ inches at the upper end and 1 inch at the lower. As the impurities rise to the surface they should be skimmed off with the greatest care, so that by the time the sirup reaches the lower end of the pan it may be perfectly clear. When the sirup reaches a temperature of 219° F.,^a or a weight of 11 pounds to the gallon, a deposit of malate of lime, or "niter," will be observed, which gradually coats the pan. This temperature and weight are proper for good sirup, which should be dipped out and carefully strained through flannel to remove the "niter." The flannel strainer is very efficacious, the sirup flowing steadily through it and coming out perfectly clear. After ten or

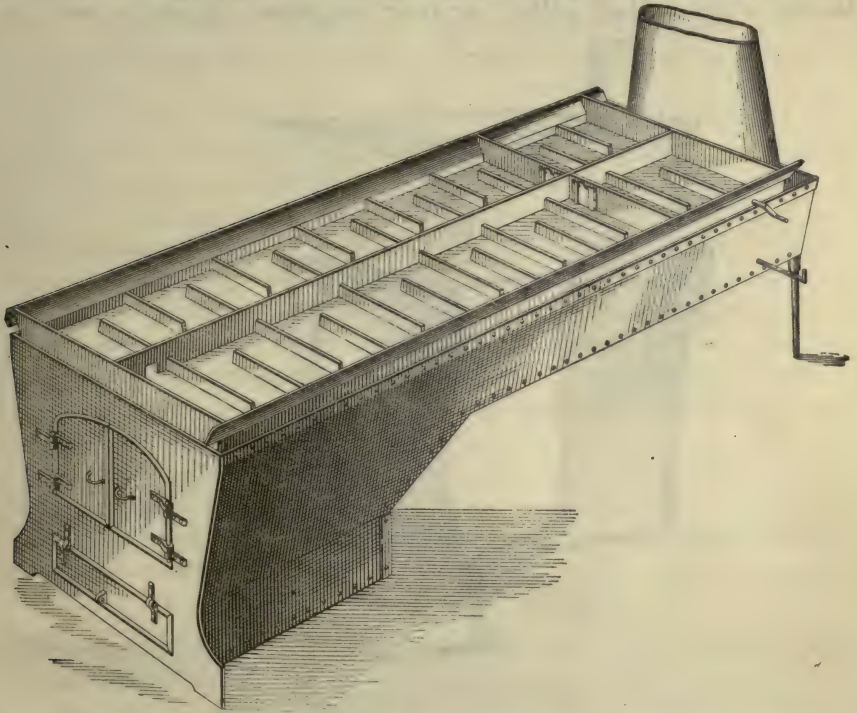


FIG. 8.—Modern maple-sap evaporator.

twelve hours' boiling, the two last sections of the evaporator will become more or less coated with the "niter" or malate of lime. Instead of scraping this off or cutting it with acids, the pan should simply be turned about, so as to put the coated sections toward the cold sap and the clean sections in the rear. In an hour or so the boiling sap will dissolve and remove the lime, all of which will be caught in the strainers. Particular care should be taken to see that the sirup finishes at

^a This figure is accepted in parts of Vermont, but since the proper temperature varies with the altitude of the sugarhouse, each operator will have to determine by experiment what is right for his situation.

the proper temperature (see footnote, p. 33) and weighs 11 pounds to the gallon. To insure this, careful tests should be made with the thermometer and the scales. The sirup should be stored in large tanks and allowed to settle, although if flannel strainers are used it will contain but little sediment.

It is a much mooted question whether the sirup should be put up for the market hot or cold. Both methods are used by experienced makers, but as it has been generally observed that sirup put up hot shows a greater tendency to crystallize, it is usually most satisfactory to put it up cold. In either case, sirup should be canned or tightly inclosed as soon as practicable. Square, oblong, or round cans containing a gallon, half gallon, or quart are used; the gallon cans are

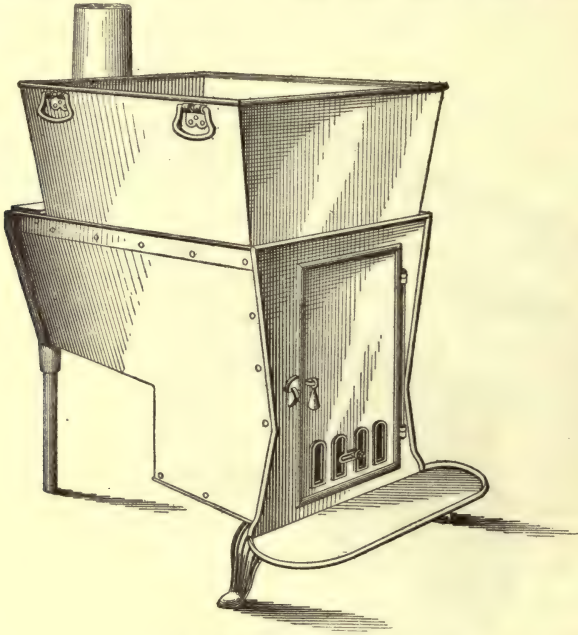


FIG. 9.—Modern sugaring-off arch.

most generally used. In filling, the can should be tipped slightly and filled to the top. It should next be lifted so that the bottom may sag and the sides bulge out slightly, and then filled to the top of its screw-cap mouth and held thus while the cork-lined screw cap is turned down as tight as possible by hand. The can should then be set down and the cap screwed on tighter with a wrench, and afterwards laid on its side to see if there is any leakage. A package containing honest measure is almost as important as sirup of good quality.

In making sugar, the sirup should be reboiled in a sugaring-off arch until it begins to crystallize, or "sugar off." By the old-fashioned methods this point was determined by pouring a little sirup on the

snow, or by dipping into it a twig bent into a loop. If the sirup became waxy on the snow, or if it formed an elastic film within the loop, it had boiled enough and was ready to "sugar." Under more modern methods the testing is done with a thermometer, and sugar is made at different temperatures according to the qualities wanted. During the early run of sap 238° F. will make cake sugar, but later in the season the sap will require 242°. If not intended for immediate consumption, or if a cake that will not "drain" is desired, the temperature may be brought up to 245° or 253° (see footnote, p. 33). At these temperatures, however, the sugar will be too hard to eat comfortably. In practice it is best to reduce the sirup to sugar in small quantities, and before pouring the mass into the molds (fig. 6, No. 1) to lower its temperature slowly by stirring, in order to avoid too rapid granulation.

The most convenient size and form for sugar is in bricks of 1 to 5 pounds and in 10-pound pails for family use. Sugar put up in bulk is likely to fall into the hands of the mixer at a low price. That in small and attractive sizes is better adapted for personal use, and more acceptable to the wholesale and retail trade. The ordinary 10-pound pail is a good package if it reaches the consumer soon after it is filled. If it is to be kept through the summer, a wooden tub or tin pail with a cover that can be hermetically sealed is better, as sugar left in an unsealed package is apt to mold in hot weather. The bricks should be wrapped in paraffin paper and packed in cases of equal lots.

Sugar and sirup should be stored in a cool, dry cellar or store-room, as excessive heat is bad for them—particularly a combination of heat and moisture, which causes the sugar to mold and the sirup to ferment.

THE PROFIT.

The production of maple sugar is so largely a home industry that normal conditions of labor and expenditure can hardly be considered. The bulk of the maple sugar on the market comes from farms where the families have supplied the labor, where the cost of the sugar grove can not be determined, and where the expenditure is entirely confined to a few utensils and a rude sugarhouse. An expenditure and profit estimate for such methods would have but little value, and could not be made specific enough to serve as an example. Nevertheless a general table can be given which will serve as a guide for the prospective maker. The following estimate is made for a grove of 15 acres upon which about 1,000 sugar trees are standing. Such woods in Vermont would cost from \$5 to \$10 per acre, according to location, but to make a perfectly safe figure the price is here taken at \$15.

Initial expenditure:

15 acres of maple woods, at \$15 per acre.....	\$225
--	-------

Equipment:

Sap buckets, 1,000, at 20 cents each.....	\$200	
Covers, 1,000, at 5 cents each.....	50	
Spouts, 1,000, at 2 cents each.....	20	
Gathering tanks, 2, at \$10 each.....	20	
Storage tank.....	20	
Sugarhouse.....	125	
Arch and evaporator.....	125	
Sugaring-off pan and arch.....	20	
Sirup cans and molds.....	12	
		592
Total.....		817

From this the annual cost may be figured as follows:

Rent of land, 6 per cent on \$225.....	\$13. 50
Wear and tear, 10 per cent of cost of equipment (\$592).....	59. 20
18 cords of firewood, at \$2 per cord.....	36. 00
3 laborers, including teamster, for twenty days, at \$1.25 per day, inclusive of board.....	75. 00
Team for hauling sap, twenty days, at \$1.25 per day.....	25. 00
Sirups cans, sugar pails, boxes, etc., for packing.....	14. 00
	222. 70

The returns, counting 3 pounds of sugar, or its equivalent in sirup, per tree, will be 3,000 pounds of sugar. At 9 cents per pound this will bring in \$270, or a net profit of \$47.30, equal to 8 per cent on \$592—the amount invested exclusive of the value of the land.

This is not a heavy return, but it must not be forgotten that the figures are purposely made conservative. Many farmers will find the labor charge, or the rent item, too high, or they may even be able to cut them out entirely. Maple sugar making is possible only at a season when farm work is usually slack, and the time devoted to it may be virtually saved. Each prospective operator will be able to make his own calculation from the figures here given, but it should be remembered that the industry has its limitations, and the number of people who can go into it will always remain comparatively small.

No attempt has been made to put into figures the probable results of improved forest methods in the sugar grove, but such considerations are believed to be of the utmost importance. This phase of the matter will receive particular attention in the experiments now being carried on by the Forest Service.

FARMERS' BULLETINS.

The following is a list of the Farmers' Bulletins available for distribution, showing the number and title of each. Copies will be sent to any address on application to any Senator, Representative, or Delegate in Congress, or to the Secretary of Agriculture, Washington, D. C.

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U. S. DEPARTMENT OF AGRICULTURE.

FARMERS' BULLETIN No. 269.

INDUSTRIAL ALCOHOL:

USES AND STATISTICS.

BY

H. W. WILEY,
CHIEF, BUREAU OF CHEMISTRY.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1906.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., October 1, 1906.

SIR: I beg to submit for publication a statement as to the uses of industrial alcohol, especially such as are of direct interest to the farmer, and some statistics relating thereto which are of value as showing the possibilities in the development of this industry. I recommend that this report be published as Farmers' Bulletin No. 269; in Farmers' Bulletin No. 268 will be found a description of the sources from which industrial alcohol can be made and the methods of manufacture. Certain text figures in the present bulletin are reproduced from drawings made from illustrations in several publications, namely: *Die Verwertung des Spiritus für technische Zwecke*; *Scientific American Supplement*, October 25, 1902; and *Spirituskraftwagen für den landwirtschaftlichen Betrieb*, to which this acknowledgment is due.

Respectfully,

H. W. WILEY,
Chief, Bureau of Chemistry.

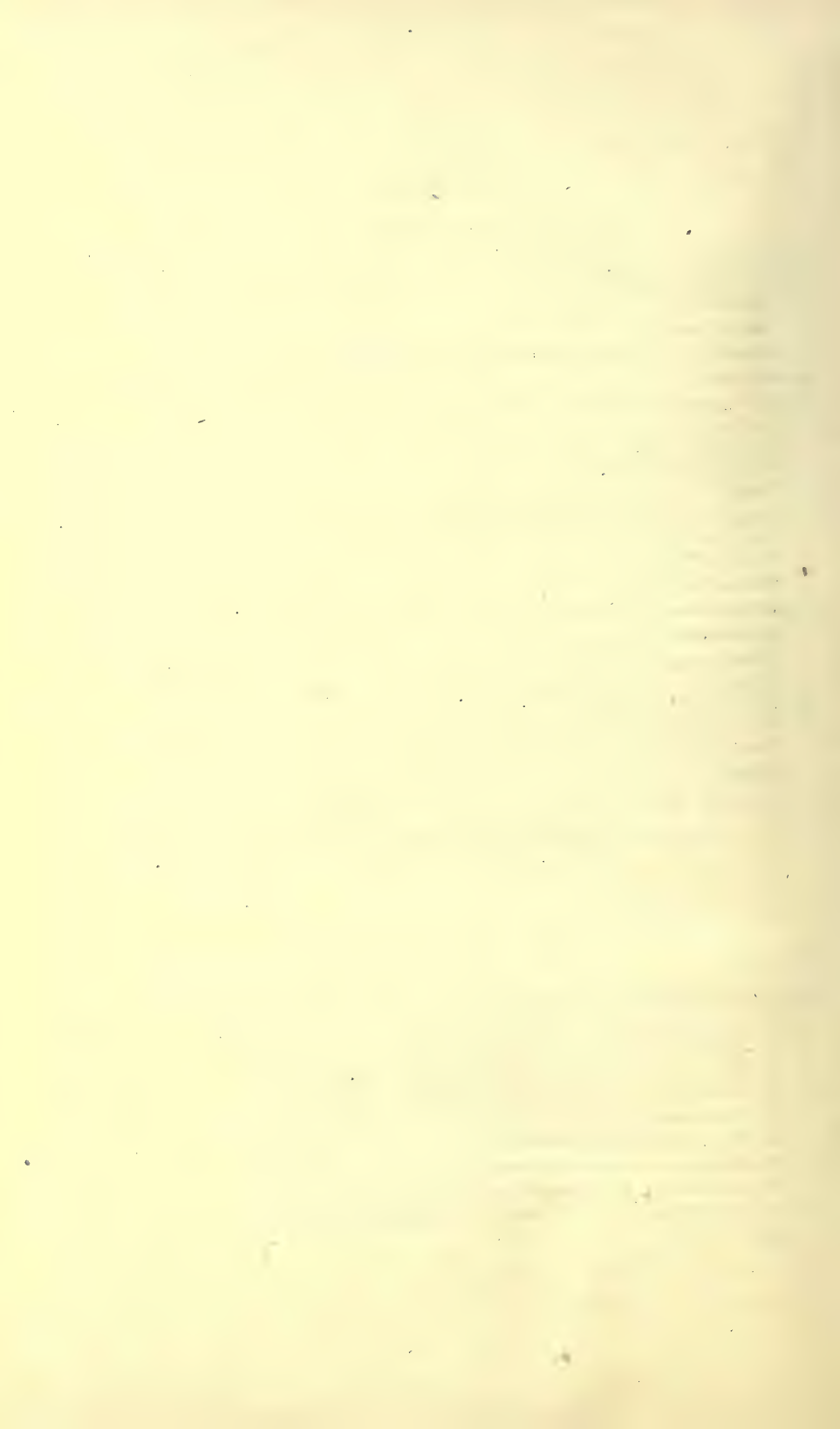
Hon. JAMES WILSON,
Secretary of Agriculture.

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INDUSTRIAL ALCOHOL: USES AND STATISTICS.

INTRODUCTION.

It is important that the farmer should have a general idea of the uses to which industrial alcohol (usually denatured) may be devoted. While this information is not necessary for the production of the raw materials, it is nevertheless valuable in indicating the particular lines of industrial development which may be promoted by free alcohol in the arts as related to the welfare of agriculture. The technical uses of alcohol are extremely numerous, and no attempt will be made to give them all, but only to mention those which are of the greatest importance.

HEATING AND ILLUMINATION.

The most important of the uses of industrial alcohol as far as the farmer is directly concerned are those included in heating and illumination. For these purposes the farmers of the country, when the processes are adjusted and the technical difficulties of production, manufacturing, and denaturing are overcome, will find alcohol extremely useful. Especially will this be true in localities remote from centers of the production of wood, coal, kerosene, gasoline, natural gas, and oil, which now are the chief heating and illuminating agents.

ALCOHOL STOVES.

The success of the alcohol stove depends largely upon the character of the wick, which must absorb the alcohol and be so adjusted as to give the necessary heating surface. By converting the alcohol into a gas and burning the vapor thus formed the wick may be dispensed with and a more effective burner obtained. The general principles involved in heating with alcohol are the same as for other heating substances. The only differences are in the methods of producing the combustion. Alcohol burns with a pale blue flame which is intensely hot. It is without smoke, and if there be any odor at all it is an agreeable and not a disagreeable one. The products of combustion of pure alcohol are water and carbon dioxide. The latter gas should be conducted out of the room by the ordinary methods of

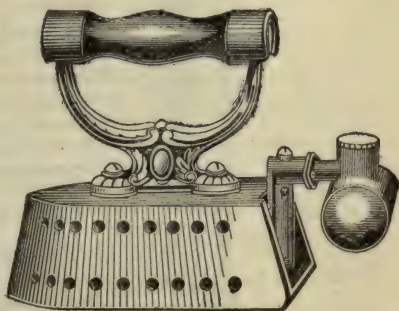


FIG. 1.—Alcohol burner for heating a flatiron.

ventilation. No form of burner should be allowed to pour the products of combustion into the room. The water which is formed is

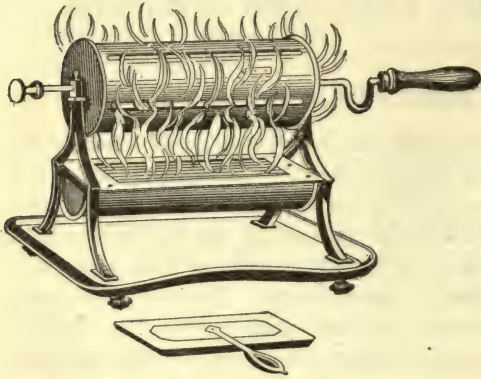


FIG. 2.—Alcohol burner for roasting coffee.

harmless, but the carbon dioxid, which is produced in large proportions, will soon vitiate the air of the room and tend to produce drowsiness, headache, and injury to health. The common methods of burning gas and kerosene in a room without ventilation are also objectionable for the same reason. Some form of ventilation by means of which the products of combustion in such cases could be removed from the room thru a chimney or otherwise is highly desirable.

could be removed from the room thru a chimney or otherwise is highly desirable.

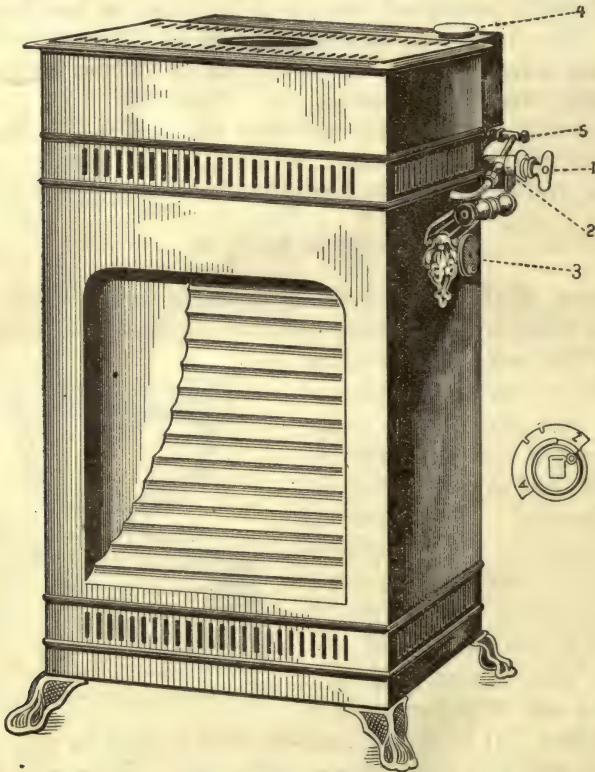


FIG. 3.—A small alcohol stove for heating purposes.

Stoves of many different kinds have been invented for burning alcohol. There are stoves for heating flatirons, soldering irons,

crimping irons, roasting coffee, etc. A smoothing iron which is designed to be heated by alcohol is shown in fig. 1, and a convenient roaster for coffee or peanuts in fig. 2.

A stove suitable for heating purposes, except that it has no chimney for carrying off the products of combustion, is pictured in fig. 3. In this figure No. 1 is the principal cock to control the admission of the gasified alcohol. No. 2 is the stopcock connecting with the heating apparatus for vaporizing the alcohol which is concealed in the mechanism of the stove. No. 3 is the stopcock for admitting a sufficient quantity of fresh alcohol before starting the stove to vaporize the alcohol for burning purposes. It is used only at the time of starting the fire, after which it is closed. No. 4 is the opening thru which the alcohol holder, which is just seen back of the stove, is filled. No. 5 is a stopcock for drawing off any remaining spirit in the holder when it is desirable to have it empty. The small figure marked *A Z* shows a

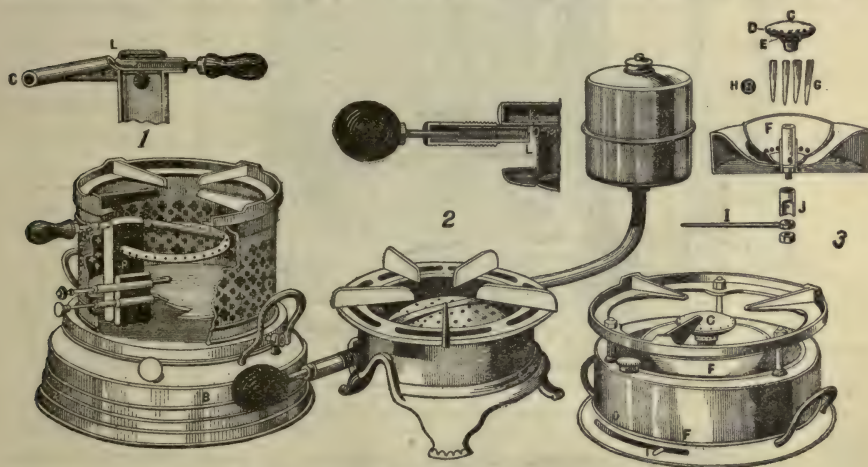


FIG. 4.—Three varieties of alcohol heaters.

device attached to the apparatus, which makes it impossible to connect the alcohol gas-forming apparatus and the stove until everything is in readiness. This is a kind of time lock upon the key, which prevents the opening or closing of the stopcocks at inopportune moments, and thus makes it impossible to flood the stove with alcohol and cause an explosion. The fluted arrangement in the front of the stove is for the purpose of reflecting the heat of the burning alcohol so as to throw it out in the largest possible quantities into the room. This is only one of the types of stove which may be used for heating purposes.

Another form of stove is shown in fig. 4, No. 1. In this stove the vaporizer is supplied by wicks *P* which draw up the alcohol from the reservoir in the bottom. A small wick (lower button at the left) serves to set the apparatus in operation by first heating the vaporizing kiln. This wick is afterwards extinguished and concealed in a

sheet which is closed by a horizontal flap (upper button). An ejector-valve *L*, represented in the upper part of the diagram, regulates the discharge of the circular burner *C*. Below the latter a white enameled disk of iron plate serves as a heat reflector. Nos. 2 and 3 are other forms of stoves which do not need any particular description.

Another form of stove is shown in fig. 5. This is a stove supplied by three circular burners, which are shown in the cross-section draw-

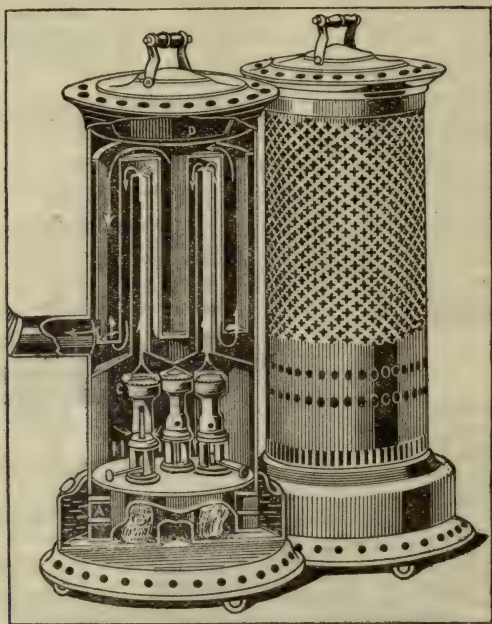


FIG. 5.—Alcohol stove with three circular burners.

ing. A double circulation of the products of combustion causes them to become separated and pass off thru a stovepipe at a relatively low temperature after having heated the air of the room as well as the vaporizer placed in the upper part of the apparatus.

ALCOHOL LAMPS.

Alcohol, as will be seen from the previous discussion respecting the alcohol stove, can not be used directly for illuminating purposes. The flame does not possess any notable illuminating power. In order that alcohol may be used for illumination it must be burned in a state of gas and the heat produced by the combustion utilized to produce incandescence in the ordinary mantle which surrounds the common gas flame for the same purpose. It has been discovered that when certain earths, such as thoria, in a state of fine subdivision, are subjected to the action of a high temperature, they become intensely white and produce by their incandescence the maximum degree of

illumination. The thoria is first deposited upon some substance such as cloth and so distributed that when the cloth burns away the particles of thoria remain in the original shape of the mantle. When held over the flame of gas or alcohol the particles become incandescent. To adjust an alcohol lamp for this purpose it is only necessary to make an attachment whereby the alcohol is first converted into a vapor. In order to light such a lamp a portion of the alcohol must first be vaporized.

In fig. 6 is shown an alcohol lamp, which is the property of the Bureau of Chemistry. The illustration shows very plainly the external mechanism of the lamp. The knob on the right is the handle of the pump, which brings a little of the alcohol around the wick holder delivering the alcohol to the vaporizing chamber. This handle is prest down two or three times by the thumb, rising by means of a spring to its original position. A few drops of alcohol are pumped up and are ignited by holding a match to the opening at the handle or above. The alcohol burns with a colorless flame for from thirty to sixty seconds. By this time the chamber is hot enough to volatilize the alcohol brought up by the wick. The mill-head on the left is then opened and the match applied at the top of the chimney, when the alcohol vapor ignites in the same manner as gas and soon heats the thorium mantle, shown inside of the chimney, to a white heat. The lamp then burns regularly without any further attention as long as any alcohol remains in the bowl.

In fig. 7 are shown two views of another lamp, the principle of which is only slightly different from that just described. The lamp on the left is cut away in order to show the internal mechanism. The alcohol is fed by the wicks *D* to the copper vaporizer *A* placed under the mantle. The burner *C* is supplied by a Bunsen burner *B* placed beneath it and having its output regulated by means of a needle valve attached to the regulator button *E*. A small lateral aperture *F* permits of the introduction of a ball of asbestos saturated with alcohol for starting the vaporization at the moment of lighting.

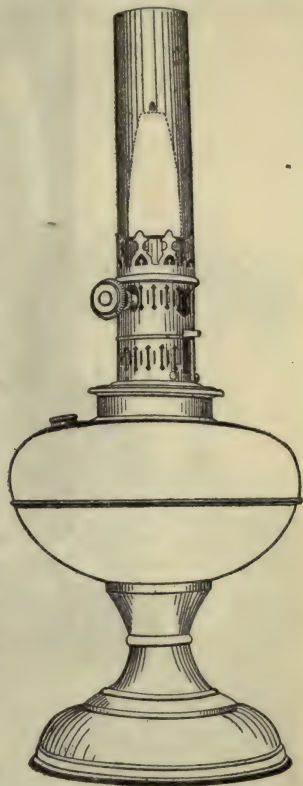


FIG. 6.—Alcohol lamp with pump attachment.

In this lamp it is noticed that the small pump for lifting the alcohol to start the vaporization is omitted, its place being taken by the ball of asbestos saturated with alcohol, inserted at the opening *F* as just stated.

It is evident that the amount of heat produced is to some extent a measure of the illuminating value when the incandescent mantle is taken into consideration. It is the high temperature which produces the incandescence and therefore the gas which in burning gives the highest temperature, other conditions being the same,

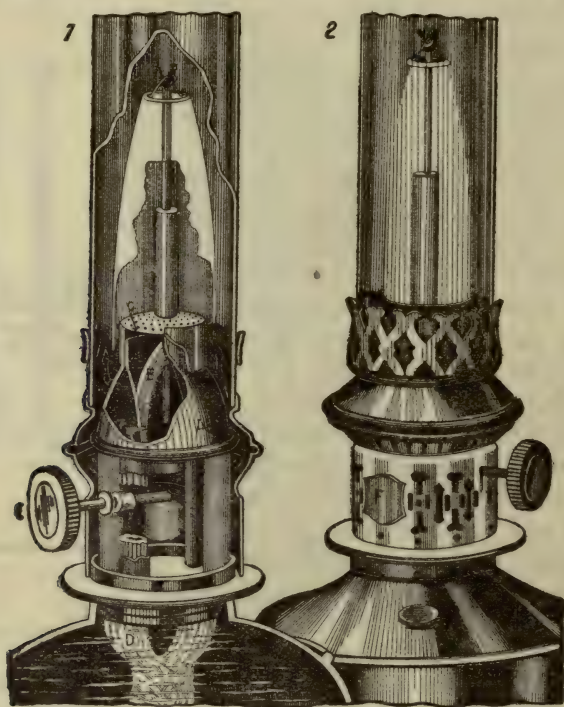


FIG. 7.—Alcohol lamp with asbestos primer.

would be of the most value for illumination. All of these points must be considered to prevent the formation of wrong opinions concerning the efficiency of alcohol for illumination, heating, and motive power, as compared with gasolene, which is the agent most used for these purposes, and which alcohol is expected to supersede.

QUANTITY OF HEAT YIELDED IN THE COMBUSTION OF ALCOHOL.

It has been estimated that 1 gram^a of gasolene will yield on complete combustion 11,000 calories,^b and 1 gram of pure alcohol 7,200 calories. No determination of the heat of combustion of ordinary

^a 1 gram = 0.03527 ounce, or 15 grains.

^b A calorie is the amount of heat necessary to raise 1 gram of water 1° centigrade (1.8° Fahrenheit).

commercial gasolene of the grade commonly used for fuel is found. Products of that nature appear to yield from 9,700 to 11,000 calories. In so far as heating purposes are concerned, therefore, it is evident that gasolene, weight for weight, is more valuable than alcohol.

ALCOHOL MOTORS.

It is quite certain that the use of alcohol motors on the farm will become quite common as soon as the technique of construction is practically complete and the price of alcohol is sufficiently low. Alcohol can be used for all purposes for which gasolene is employed, namely, the driving of wagons, carriages, stationary motors, water



FIG. 8.—Adaptation of an alcohol motor for plowing.

pumps, mowing machines, plows, etc. Very little change need be made in the engine of a motor car designed to use gasolene to fit it for the use of alcohol. Gasolene becomes volatile at a temperature of blood heat (98.5° F.), while a much greater degree of heat (158° to 176° F.) is necessary to volatilize alcohol rapidly enough for motor purposes. This fact makes necessary a change in the explosion chamber of the engine when alcohol is to be used. This adjustment is especially important in the starting of the machine, as after it is in action the

temperature of combustion is quite sufficient to easily produce the gasification necessary.

Fig. 8 represents an ordinary plow suitable for attaching to a heavy motor apparatus driven by alcohol; fig. 9, a mowing machine, and fig. 10, a reaping and binding machine, both driven by alcohol motors. In regard to these heavy machines it may be said that they probably would come into use only on large farms where the surface of the soil is practically level. They would not be suitable for small farms nor those in hilly sections. In this connection attention is called to the fact that steam plowing, altho practicable and profitable under certain conditions, has not been practised to any great extent in this country, in fact, not nearly so much as in England. It is not likely, therefore, that plowing and harvesting by alcohol motors will come into use very soon, altho the possibilities are worthy of the consideration of the thoughtful farmer. On the other hand, it seems probable



FIG. 9.—A mowing machine propelled by an alcohol motor.

that small motors for driving machines for chopping and grinding cattle food, pumping water, and similar purposes may be brought into very general use as soon as the denatured alcohol becomes cheap enough to render its use practicable.

In the driving of motor engines the quantity of heat evolved is not always a measure of efficiency. It is not the purpose of this bulletin to go into this subject at all, only to say that the vapor of alcohol can be more highly compressed at any given temperature without exploding than can the vapor of gasoline. As the decreased volume of the mixture of the explosive vapor and air is to a certain extent a measure of efficiency when engines are driven by the expansion of gases, the high degree of compressibility of the alcohol vapor without danger of explosion may compensate for the smaller quantity of heat which is generated by its combustion.

The problems connected with the use of alcohol for driving machinery are somewhat technical, and it is only desired to call attention to the possible advantages to the farmer from this source of power, and also to point out the difficulties which must be overcome. In this connection it seems that a word of caution is needed, as in the exploitation of tax-free alcohol extravagant opinions regarding its possibilities have been expressed. These exaggerated statements have been made without any intent to deceive or mislead, but on account of insufficient information. The natural tendency in all such matters is to select those points which are certain to be of great benefit and publish them broadcast, and to neglect the diffi-



FIG. 10.—Adaptation of an alcohol motor to a reaper and binder.

culties and dangers which lie in the path of progress along these lines. Our farmers, who are naturally conservative, need very little caution in such matters, but it is important that a full understanding of the difficulties of these problems should be disseminated among the agricultural population. It is quite certain that if alcohol can be produced in the near future at a cost not exceeding 25 or 30 cents per gallon of 95 per cent strength, it will be a most valuable source of power on the farm. Altho with the present relative prices of alcohol and gasolene there is no financial advantage in the use of the former, it is highly probable that the price of gasolene will advance and that of alcohol fall. Thus the farmhouse and the

barn may be liberally supplied with water at such an elevation that it can be used with all the facility enjoyed by those who live in the city by means of a safe, cheap, and effective method of pumping made possible by the alcohol motor. The machinery around the barn and the stables which is utilized for chopping food and grinding grain in the preparation of rations for domesticated animals should be of a character which is efficient and at the same time without danger. An alcohol motor placed in a small room separated from the barn at such a distance as not to endanger it in case of an accident would make it possible to supply power of this kind. Altho alcohol is far less dangerous in use than gasolene as far as probability of explosion is concerned, there should be no misunderstanding respecting the fact that it is an explosive substance both when in the form of vapor and when mixt with air, and all the precautions which are used in the case of gasolene should be employed also with alcohol. While the use of these precautions will practically eliminate any source of danger, it is nevertheless advisable, even in the case of alcohol, to separate the building in which it is used from the barn, which contains more or less highly combustible matter. The fact that a substance is less dangerous than another is no excuse for omitting any of the precautions to prevent injury as the result of accident.

USES OF DENATURED ALCOHOL NOT DIRECTLY ENTERING INTO FARM OPERATIONS.

It seems advisable that some of the uses of industrial alcohol not directly connected with farm operations should be known to the farmer, in order that he may be fully informed respecting the industry in which he necessarily takes so important a part. The purposes for which tax-free alcohol can be used in the arts are fully set forth in a public document entitled "Free Alcohol, Hearings before the Committee on Ways and Means, House of Representatives, Fifty-ninth Congress, first session, February-March, 1906." A very full discussion of the subject is also found in an English report entitled "Industrial Alcohol Committee, Minutes of Evidence Taken before the Departmental Committee on Industrial Alcohol, with Appendices. Presented to both Houses of Parliament by Command of His Majesty. Printed for His Majesty's Stationery Office by Wyman & Sons (Limited), Fetter Lane, London, E. C., 1905." The evidence submitted in the two reports mentioned contains practically all that is known concerning the uses of denatured alcohol. It is not intended here even to make a résumé of this evidence; only the most important industries which are benefited by tax-free alcohol can be mentioned.

COAL-TAR COLORS.

Alcohol is used very extensively in the manufacture of dyes and other by-products from coal tar. The utilization of coal tar is an industry which has been almost exclusively monopolized by Germany, owing, in the opinion of many, to the fact that the Germans early recognized the importance of using alcohol free of tax in such industries. Whether or not this is the case may be open to some question, but it appears probable that this industry can not be established and flourish in the United States without the advantage of untaxed alcohol.

SMOKELESS POWDER.

The manufacture of smokeless powder is one of the industries in which tax-free alcohol is of the highest importance. Smokeless powder consists essentially of cotton which is subjected to treatment with sulfuric and nitric acids, whereby a certain quantity of oxid of nitrogen is introduced into the cotton, altering its properties without changing its appearance. The cotton thus treated is entirely different in character from the natural product. It burns with great ease and, when confined, with explosive violence, giving off practically no smoke and but few noxious fumes; hence for these two reasons it is especially valuable. Before use it is necessary that it be dissolved or at least reduced to a homogeneous, pasty mass. For this purpose the solvents which are most employed are ether and alcohol. Ether itself, as will be seen further on, is one of the products made from alcohol, and hence alcohol becomes a dominant factor in the manufacture of smokeless powder. If smokeless powder must be made with alcohol on which the tax has been paid it can not be profitably distributed for perhaps less than \$1.25 or even more per pound. If tax-free alcohol can be used the price can be reduced more than one-half. In the smokeless powder made for Government use under the present regulations the alcohol which is employed is allowed to be used free of tax under supervision and thus the Government secures the powder at a much lower rate than would otherwise be the case. This, however, makes no difference to the Government, since it might collect the tax on the alcohol and pay the larger price per pound without any loss whatever. For the private consumer of powders the case is quite different, as he is compelled to pay the higher price when tax-free alcohol can not be used.

VARNISHES, LACQUERS, ETC.

One of the most important technical uses of alcohol is in the manufacture of varnishes and lacquers, where the gums which are employed are necessarily dissolved in alcohol. This use of alcohol is extremely important and affects a great many industries.

ETHER.

The ether of commerce, sometimes called sulfuric ether, is manufactured exclusively from alcohol by the action of sulfuric acid and heat. This ether is used in a great many technical operations, since it is one of the best solvents known, especially for fats. It is also extensively used in surgery as an anesthetic. Under the present arrangements ether used for technical purposes can only be made from alcohol on which tax has been paid, and thus its price is phenomenally high. By the use of tax-free alcohol for making ether, this price would be proportionally reduced, and in some countries the law permits the use of alcohol denatured by a special formula for this purpose. The denaturation of alcohol by the general process prescribed by the Commissioner of Internal Revenue^a may unfit it for the manufacture of ether, even if such use were deemed admissible under the new law^b and the regulations made in conformity therewith.

MEDICINES AND PHARMACEUTICAL PREPARATIONS.

Alcohol is used very extensively in the preparation of medicines. That great body of remedies known as tinctures is made by using alcohol as a solvent for the active principle of the herbs and plants from which the tinctures are made. The law, however, does not permit the use of denatured alcohol for "liquid medicinal purposes," and, concerning preparations which are used both in pharmaceutical preparations and in the arts, it is to be noted that under section 80 of the Regulations the Commissioner of Internal Revenue will consider all formulas submitted for the special denaturing of alcohol for technical purposes and will determine whether their use is consistent with the proper safeguarding of the revenue. The regulation states further: "But one special denaturant will be authorized for the same class of industries, unless it shall be shown that there is good reason for additional special denaturants."

IMITATION SILK.

The substance which is known as imitation silk is really a production from cotton or other cellulose material which, in its finished state, resembles silk somewhat in luster. It is not silk, and hence

^a The formula for completely denaturing alcohol given by the regulations of the United States Internal Revenue is as follows: To 100 parts of ethyl alcohol add 10 parts of approved methyl alcohol and one-half of 1 part of approved benzin. Formulas for special denaturants for specific purposes are to be submitted by manufacturers to the Commissioner of Internal Revenue, who will announce from time to time what formulas may be used in the several classes of industries.

^b For the text of the law permitting the use of denatured alcohol, tax-free, for industrial purposes, see Farmers' Bulletin 268, Industrial Alcohol, Sources and Manufacture.

not even artificial silk. It is a textile product which has the promise of a successful future and is therefore of interest not only to the manufacturer and the consumer but to the farmer who produces the cellulose. Imitation silk is in a measure the same substance as smokeless powder, except that after it is made the nitrogenous constituents are removed, so as to restore the finished product again to the condition of ordinary cotton, devoid of explosive properties. In the making of imitation silk a partial nitrification of the cotton is accomplished in much the same manner as in making smokeless powder. The partially nitrated cotton is then reduced to a paste by solution in alcohol, ether, or other solvent, and in this condition is forced thru small orifices, producing fine fibers of a silky luster. After these are produced the oxid of nitrogen is removed from them by a chemical process which does not interfere with their lustrous appearance. These fibers are then spun and woven as ordinary silk or cotton, producing fabrics which resemble in luster the natural silk. The utilization of tax-free alcohol would be a great stimulus to the advancement of industries of this class.

ARTIFICIAL VINEGAR.

Dilute alcohol, commonly known as low wines, can be utilized for the manufacture of vinegar. For this purpose the dilute alcohol is made to pass over the fresh shavings of beech wood. These beech shavings furnish the organisms which oxidize the alcohol into acetic acid, and after passing thru a series of vats containing these shavings the alcohol is converted into a dilute solution of acetic acid. During the process there is a considerable loss of alcohol by evaporation. It is estimated that only about 70 per cent of alcohol is finally recovered as vinegar. This material ought not to be called vinegar, as it is acetic acid pure and simple. It is colored and treated so as to resemble vinegar made from cider or wine or malt, and to this extent becomes an adulteration. This industry may be mentioned as one that would be promoted by the use of tax-free alcohol, altho it deserves little if any encouragement. Section 59 of the Regulations concerning denatured alcohol, however, especially names "manufacturers of vinegar by the vaporizing process" among the classes of persons who may not store denatured alcohol on their premises or make use of it.

FLAVORING EXTRACTS.

The flavoring extracts of commerce are made largely with alcohol as a solvent. This should be the purest possible alcohol, and since it can not possibly be denatured and still remain potable and attractive, the law would probably not apply under conditions of this kind.

USE OF UNDENATURED ALCOHOL FREE OF TAX.

Under laws other than the denatured alcohol law, alcohol may be withdrawn from the distillery warehouse free of tax for certain purposes. Among these may be mentioned its use by the United States Government, its use for scientific purposes under certain conditions, and its use in the manufacture of sugar from sorghum. During the year ended June 30, 1905, 2,112,830.9 gallons of spirits were withdrawn for these purposes. Details in regard to such withdrawals can be obtained from the Commissioner of Internal Revenue, whose annual report for the year ended on the date mentioned furnishes valuable information for those proposing to engage in any way in the distillation of alcohol.

Attention should also be called to the fact that large quantities of undenatured alcohol are manufactured in the United States on which no tax is paid. The principal sources of this alcohol are as follows:

HARD CIDER.

The natural conversion of sweet cider into hard cider by the ferments which it contains is a process in which large quantities of alcohol are produced and on which no tax is paid. The term alcohol when used alone does not apply to such products, but only to alcohol produced by distillation. As is well known, the sugar which is one of the principal constituents of fresh cider is mixt with yeasts which naturally attach to apples, so that when exposed without sterilization fermentation takes place, the sugar disappears, and alcohol is formed. If we assume that the average apple juice contains 12 per cent of fermentable matter, it is seen that about 6 per cent of alcohol may be developed in a hard cider. No tax is put upon this cider, nor is there any supervision on the part of the Commissioner of Internal Revenue in its production.

WINE.

In the manufacture of wine the natural yeasts which attach to the grapes produce in the exprest grape juice a fermentation by means of which the grape juice is converted into wine. This is a vast industry in many countries and a very important industry in the United States. The alcohol which is formed in this way pays no tax, nor does the manufacturer of ordinary wines conduct his business under the supervision of the Commissioner of Internal Revenue. What is true of the juices of the grape and the apple is true of other fruit juices. They may be all of them fermented and their sugar converted into alcohol without Government supervision and without paying any tax. But when the alcohol which is produced by the fermentation of fruit juices is subjected to distillation

it comes under the control of the Commissioner of Internal Revenue. The distillation of alcohol from waste products is conducted under special regulations authorized by law.

SWEET WINE.

Brandy and other distilled spirits made from grapes or their refuse may be used for fortifying sweet wines upon the payment of a nominal tax of 3 cents a gallon, as provided for by the following act of Congress, which indicates the character of the wines that may be fortified in this way:

AN ACT To amend existing laws relating to the fortification of pure sweet wines.

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That section forty-three of the Act entitled "An Act to reduce the revenue and equalize duties on imports, and for other purposes," approved October first, eighteen hundred and ninety, as amended by section sixty-eight of the Act of August twenty-seventh, eighteen hundred and ninety-four, be further amended, so as to read as follows:

SEC. 43. That the wine spirits mentioned in section forty-two of this Act is the product resulting from the distillation of fermented grape juice, to which water may have been added prior to, during, or after fermentation, for the sole purpose of facilitating the fermentation and economical distillation thereof, and shall be held to include the product from grapes or their residues, commonly known as grape brandy; and the pure sweet wine, which may be fortified free of tax, as provided in said section, is fermented grape juice only, and shall contain no other substance whatever introduced before, at the time of, or after fermentation, except as herein expressly provided; and such sweet wine shall contain not less than four per centum of saccharine matter, which saccharine strength may be determined by testing with Balling's saccharometer or must scale, such sweet wine, after the evaporation of the spirits contained therein, and restoring the sample tested to original volume by addition of water: *Provided*, That the addition of pure boiled or condensed grape must or pure crystallized cane or beet sugar or pure anhydrous sugar to the pure grape juice aforesaid, or the fermented product of such grape juice prior to the fortification provided by this Act for the sole purpose of perfecting sweet wines according to commercial standard, or the addition of water in such quantities only as may be necessary in the mechanical operation of grape conveyors, crushers, and pipes leading to fermenting tanks, shall not be excluded by the definition of pure sweet wine aforesaid: *Provided, however*, That the cane or beet sugar, or pure anhydrous sugar, or water, so used shall not in either case be in excess of ten per centum of the weight of the wine to be fortified under this Act: *And provided further*, That the addition of water herein authorized shall be under such regulations and limitations as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may from time to time prescribe; but in no case shall such wines to which water has been added be eligible for fortification under the provisions of this Act where the same, after fermentation and before fortification, have an alcoholic strength of less than five per centum of their volume.

SEC. 2. That section forty-nine of the said Act, approved October first, eighteen hundred and ninety, be amended so as to read as follows:

SEC. 49. That wine spirits used in fortifying wines may be recovered from such wine only on the premises of a duly authorized grape-brandy distiller; and for the purpose of such recovery wine so fortified may be received as material on the premises of such a distiller, on a special permit of the collector of internal revenue in whose district the distillery is located; and the distiller will be held to pay the tax on a product from such wines as will include both the alcoholic strength therein produced by the fermentation of the grape juice and that obtained from the added distilled spirits, subject, however, to the provisions of section

thirty-three hundred and nine of the Revised Statutes of the United States, as amended by section six of the Act entitled "An Act to amend the laws relating to internal revenue," approved March first, eighteen hundred and seventy-nine; and such spirits so recovered may be used by such distiller to fortify wines as authorized by section forty-two of the aforesaid Act, approved October first, eighteen hundred and ninety.

SEC. 3. That the Commissioner of Internal Revenue is hereby authorized to assign at each winery where wines are to be fortified such number of gaugers or storekeeper gaugers, in the capacity of gaugers, for special duties as may be necessary for the proper supervision of the making and fortifying of such wines, and the compensation of such officers shall not exceed five dollars per diem while so assigned, together with their actual and necessary traveling expenses, and also a reasonable allowance for their board bills, to be fixed by the Commissioner of Internal Revenue, but not to exceed two dollars per day for said board bills; and to cover the expenses to the Government attending the making and fortification of such sweet wines there shall be levied and assessed against each maker of such wines, and collected monthly, a charge of three cents on each taxable gallon of brandy used by him in the fortification of such wines during the preceding month. That bonds hereafter given under the provisions of the aforesaid Act of October first, eighteen hundred and ninety, as amended, shall be conditioned for the payment of the tax on all brandy removed thereunder and not used and accounted for within the time and in the manner required by law and regulations, and for the payment of all charges herein imposed on the brandy so withdrawn and used; and the said bond shall contain such other conditions as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may by regulation prescribe.

SEC. 4. That where brandy to be used in the fortification of wine is distilled on premises adjacent to the winery premises the Commissioner of Internal Revenue may, in his discretion, authorize the erection on either of said premises of fermenting vats for material to be used either in the manufacture of such wines or the brandy to be used in the fortification thereof; and all such materials used or received on either of said premises shall be under the supervision of the officer assigned to such winery, and shall be accounted for at such times and in such manner as the Commissioner may direct.

SEC. 5. That the provisions of sections thirty-two hundred and twenty-one and thirty-two hundred and twenty-three of the Revised Statutes of the United States, as amended by an Act approved March first, eighteen hundred and seventy-nine, are hereby extended to grape brandy withdrawn for use in the fortification of sweet wines, and which, prior to such use, is accidentally destroyed by fire or other casualty while stored in the fortifying room on the winery premises.

SEC. 6. That any person who by any process recovers from wines fortified under the provisions of the aforesaid Act approved October first, eighteen hundred and ninety, or amendments thereto, any brandy or wine spirits used in the manufacture or fortification of said wine, otherwise than is provided for in said Act and its amendments, or who shall rectify, mix, or compound with other distilled spirits such fortified wines or grape brandy or wine spirits unlawfully recovered therefrom, shall, on conviction, be punished for each such offense by a fine of not less than two hundred dollars nor more than one thousand dollars. But the provisions of this section, and the provisions of section thirty-two hundred and forty-four of the Revised Statutes of the United States, as amended, relating to rectification, shall not be held to apply to the blending of pure sweet wines fortified under the provisions of the said Act of October first, eighteen hundred and ninety, or amendments thereto, where such wines are blended for the sole purpose of perfecting the same according to commercial standard.

The following table shows the extent of the operations under this law, the kinds of sweet wines so fortified including angelica, concord, malaga, muscatel, port, scuppernong, sherry, sweet catawba, and tokay:

TABLE I.—Quantity of grape brandy withdrawn from distilleries and bonded warehouses free of tax, and quantity of wines so fortified, by districts, for the year ended June 30, 1905.^a

District.	Grape brandy used in forti- fication.	Total wines produced.	
		Before forti- fication.	After forti- fication.
	<i>Gallons.</i>	<i>Gallons</i>	<i>Gallons.</i>
First California.....	2,661,912.5	9,179,258.47	10,716,640.78
Fourth California.....	598,509.5	2,083,922.13	2,444,548.65
First Missouri.....	272.4	873.48	1,024.06
First New Jersey.....	714.0	5,450.00	6,150.50
Twenty-eighth New York.....	131,438.7	447,113.07	522,757.84
Fourth North Carolina.....	2,308.7	36,049.00	37,747.00
Tenth Ohio.....	11,374.5	39,788.50	46,678.00
Second Virginia.....	24,289.3	200,029.48	214,500.00
Total.....	3,430,819.6	11,992,554.13	13,990,055.83

^a Annual Report of the Commissioner of Internal Revenue, 1905, p. 131.**STATISTICS ON ALCOHOL PRODUCTION.****THE UNITED STATES.**

To give an idea of the magnitude of the present industry in distilled spirits and the possibilities of its enlargement, the following statistical data are quoted from the report of the Commissioner of Internal Revenue for the fiscal year ended June 30, 1905:

TABLE II.—Distilleries registered and operated during the year ended June 30, 1905.

State or Territory.	Grain.		Molasses.		Fruit.		Total regis- tered.	Total oper- ated.
	Regis- tered.	Oper- ated.	Regis- tered.	Oper- ated.	Regis- tered.	Oper- ated.		
Alabama.....	17	15			17	17	34	32
Arkansas.....	25	16			13	12	38	28
California.....	1	1	2	1	224	218	227	220
Colorado.....	2	2			1	1	3	3
Connecticut.....	3	3			28	23	31	26
Delaware.....	1				10	10	11	10
Florida.....					1	1	1	1
Georgia.....	40	39			32	32	72	71
Idaho.....					3	3	3	3
Illinois.....	12	10			5	5	17	15
Indiana.....	22	11			17	16	39	27
Iowa.....					1	1	1	1
Kansas.....					1		1	
Kentucky.....	257	223	1	1	30	29	288	253
Louisiana.....	1		2	2	3	3	6	5
Maryland.....	30	25			10	8	40	33
Massachusetts.....	1	1	6	6	3	2	10	9
Michigan.....			1	1			1	1
Missouri.....	61	43			18	18	79	61
Nebraska.....	1	1			1	1	2	2
New Hampshire.....	1		1	1			2	1
New Jersey.....	1				36	34	37	34
New Mexico.....					3	3	3	3
New York.....	5	2	1	1	36	32	42	35
North Carolina.....	66	57			326	298	392	355
Ohio.....	29	23			30	26	56	49
Oklahoma.....	7	3					7	3
Oregon.....					5	5	5	5
Pennsylvania.....	100	83			9	8	109	91
Rhode Island.....	1	1			3	3	4	4
South Carolina.....	14	9					14	9
Tennessee.....	70	58			28	24	98	82
Texas.....	2	1			8	8	10	9
Utah.....					1	1	1	1
Virginia.....	111	80			198	182	309	271
Washington.....	2				3	3	5	3
West Virginia.....	8	8			4	4	12	12
Wisconsin.....	5	4					5	4
Total.....	896	728	14	13	1,108	1,031	2,018	1,772

TABLE III.—Quantities of grain and other materials used for the production of distilled spirits during the fiscal year ended June 30, 1905, by States and Territories.

State or Territory.	Malt.	Wheat.	Barley.	Rye.	Corn.
	<i>Bushels.</i>	<i>Bushels.</i>	<i>Bushels.</i>	<i>Bushels.</i>	<i>Bushels.</i>
Alabama.....	5,580		28	3,443	30,978
Arkansas.....	1,637		11	735	11,034
California.....	741	1,188	2,396	2,169	1,288
Colorado.....	20			66	171
Connecticut.....	10,834			20,106	17,266
Georgia.....	13,000			483	67,942
Illinois.....	1,112,069			315,912	8,053,364
Indiana.....	462,695	183		119,321	3,959,267
Kentucky.....	888,279	5,373	3,920	1,206,595	4,670,252
Maryland.....	250,667			1,180,991	29,552
Massachusetts.....	2,093			6,329	5,885
Missouri.....	14,153	478		24,810	59,670
Nebraska.....	47,327			13,801	334,687
New York.....	139,012			334,293	342,712
North Carolina.....	25,376			38,127	183,374
Ohio.....	288,879	3,858		364,587	1,990,599
Oklahoma Territory.....	275			307	2,763
Pennsylvania.....	372,680	1,029	314	1,595,721	111,627
Rhode Island.....	231			445	410
South Carolina.....	18,453			24,918	113,323
Tennessee.....	37,206	58	232	28,735	249,523
Texas.....	17				186
Virginia.....	12,989	314		31,445	55,464
West Virginia.....	16,960			65,132	2,996
Wisconsin.....	77,415		2,973	110,557	298,171
Total.....	3,798,578	12,481	9,874	5,489,028	20,592,504

State or Territory.	Oats.	Mill feed.	Molasses.	Other materials.	Total.	
	<i>Bushels.</i>	<i>Bushels.</i>	<i>Gallons.</i>	<i>Bushels.</i>	<i>Bushels.</i>	<i>Gallons.</i>
Alabama.....					40,029	
Arkansas.....					13,407	
California.....			1,378,957		7,782	1,378,957
Colorado.....					257	
Connecticut.....					48,206	
Georgia.....					81,425	
Illinois.....	595				9,481,850	
Indiana.....	2,787				4,544,253	
Kentucky.....	2,489	21	219,996		6,776,929	219,996
Louisiana.....			7,036,457			7,036,457
Maryland.....	1,414				1,462,624	
Massachusetts.....			1,923,682		14,307	1,923,682
Michigan.....			4,419,716			4,419,716
Missouri.....		109			99,220	
Nebraska.....					395,815	
New Hampshire.....			18,225			18,225
New York.....			5,545,200		816,017	5,545,200
North Carolina.....					246,877	
Ohio.....	11,703	200			2,659,826	
Oklahoma Territory.....					3,345	
Pennsylvania.....			7,320		2,081,371	7,320
Rhode Island.....					1,086	
South Carolina.....					156,694	
Tennessee.....		1,132			316,886	
Texas.....					203	
Virginia.....					100,212	
West Virginia.....					85,088	
Wisconsin.....				4,500	493,616	
Total.....	18,898	1,462	20,549,553	4,500	29,927,325	20,549,553

The average yield per bushel of grain was $\frac{133,932,478}{29,927,325} = 4.47+$ gallons of spirits.

The average yield per gallon of molasses used for the production of spirits was $\frac{12,086,329}{18,387,650} = 0.657+$ gallon.

The average yield per gallon of molasses used for the production of rum was $\frac{1,791,987}{2,161,903} = 0.828+$ gallon.

TABLE IV.—Quantity of each kind of fruit brandy produced during the fiscal year ended June 30, 1905, by collection districts.

District.	Apple.	Peach.	Grape.	Pear.	Apricot.
	Gallons.	Gallons.	Gallons.	Gallons.	Gallons.
Alabama.....	2,609.8	702.6	7.0		
Arkansas.....	26,531.0	725.5	66.0		
First California.....	441.0	18,068.9	3,811,625.7	2,904.6	1,466.3
Fourth California.....			790,508.5		1,758.8
Connecticut.....	17,812.6		1,876.9		
Florida.....			16.0	144.8	
Georgia.....	3,569.4	3,532.5	41.3	14.0	
Fifth Illinois.....			133.0		
Eighth Illinois.....			183.2		
Sixth Indiana.....	551.8	68.4	74.0		
Seventh Indiana.....	68,507.1	30.0			
Fourth Iowa.....			28.5		
Second Kentucky.....	12,376.1				
Fifth Kentucky.....	86,745.7				
Seventh Kentucky.....	732.0				
Eighth Kentucky.....	810.9				
Louisiana.....		9.4			
Maryland.....	17,206.4	20,662.6		5,019.5	
Third Massachusetts.....	2,135.8				
First Missouri.....	886.2	966.3	7,408.1		
Sixth Missouri.....	970.6	445.0	363.4		
Montana.....	227.7	150.2			
Nebraska.....	15.4				
First New Jersey.....	56,502.8		285.0		
Fifth New Jersey.....	56,850.3	86.6	2,291.1		
New Mexico.....	25.0		140.9		
First New York.....	217.0		1,713.6		
Fourteenth New York.....	22,678.3				
Twenty-first New York.....	133,501.0				
Twenty-eighth New York.....	20,752.4	30.5	25,476.5		
Fourth North Carolina.....	10,021.9	72.5	1,055.0		
Fifth North Carolina.....	25,035.6	150.0			
First Ohio.....	1,713.1		119.0		
Tenth Ohio.....	8,959.9	1,498.7	72,371.3		
Eleventh Ohio.....	433.0		142.5		
Eighteenth Ohio.....	1,052.6		977.5		
Oregon.....	116.6	17.0			
First Pennsylvania.....	15,646.3		355.9		
Second Tennessee.....	210.0				
Fifth Tennessee.....	19,745.0	175.0			
Fourth Texas.....		530.4			
Second Virginia.....	3,974.2	2.5	2,706.8		
Sixth Virginia.....	11,251.6	340.0	463.5		
Washington.....	218.2	129.5			
West Virginia.....	1,309.4				
Total.....	632,343.7	48,304.1	4,720,430.2	8,082.9	3,226.1

TABLE IV.—Quantity of each kind of fruit brandy produced during the fiscal year ended June 30, 1905, by collection districts—Continued.

District.	Berry.	Prune.	Fig.	Cherry.	Total.
	Gallons.	Gallons.	Gallons.	Gallons.	Gallons.
Alabama.....					3,319.4
Arkansas.....					27,322.5
First California.....		24,439.9			3,858,946.4
Fourth California.....		1,927.5			794,194.8
Colorado.....			61.8		61.8
Connecticut.....		214.3	201.5		20,105.3
Florida.....					160.8
Georgia.....					7,157.2
Fifth Illinois.....					133.0
Eighth Illinois.....					183.2
Sixth Indiana.....					694.2
Seventh Indiana.....					68,537.1
Fourth Iowa.....					28.5
Second Kentucky.....					12,376.1
Fifth Kentucky.....					86,745.7
Seventh Kentucky.....					732.0
Eighth Kentucky.....					810.9
Louisiana.....					9.4
Maryland.....					42,888.5
Third Massachusetts.....					2,135.8
First Missouri.....					9,260.6
Sixth Missouri.....		55.0		101.6	1,935.6
Montana.....		21.8			399.7
Nebraska.....					15.4
First New Jersey.....		1,782.2			58,570.0
Fifth New Jersey.....		284.2	146.5	20.5	59,679.2
New Mexico.....					165.9
First New York.....		1,486.2	305.6		3,722.4
Fourteenth New York.....					22,678.3
Twenty-first New York.....					133,501.0
Twenty-eighth New York.....	32.5				46,291.9
Fourth North Carolina.....	12.0				11,161.4
Fifth North Carolina.....					25,185.6
First Ohio.....					1,832.1
Tenth Ohio.....		2,456.0			85,285.9
Eleventh Ohio.....					575.5
Eighteenth Ohio.....	726.4				2,756.5
Oregon.....		421.9		15.0	570.5
First Pennsylvania.....		1,099.4			17,101.6
Second Tennessee.....					210.0
Fifth Tennessee.....					19,920.0
Fourth Texas.....					530.4
Second Virginia.....	95.0				6,778.5
Sixth Virginia.....				10.0	12,065.1
Washington.....		191.6			539.3
West Virginia.....					1,309.4
Total.....	865.9	34,380.0	715.4	147.1	5,448,584.4

TABLE V.—Comparative statement of materials used and spirits produced during the last two fiscal years.

Year.	Grain used.	Spirits produced from grain.	Molasses used to produce spirits.	Spirits produced from molasses.	Molasses used to produce rum.	Rum produced.	Total production.
	Bushels.	Gallons.	Gallons.	Gallons.	Gallons.	Gallons.	Gallons.
1904.....	27,687,683	123,549,427	16,425,853	8,961,346	2,123,553	1,801,179	134,311,952
1905.....	29,927,325	133,932,478	18,387,650	12,086,329	2,161,903	1,791,987	147,810,794

GREAT BRITAIN.

The new law relating to the denaturing of alcohol in England for industrial purposes went into effect on October 1, 1906. One of the principal objects of the law is to extend the uses of denatured alcohol to the manufacture of certain products which could not be made with the ordinary denatured alcohol as provided under the old law. Under the new order denatured alcohol may be used for the following additional purposes:

1. Methylated spirits may be used in the manufacture of sulfuric ether, ethyl chlorid, methyl chlorid, ethyl bromid, chloroform, and hydrate of chloral, for use as a medicine or in any art or manufacture; and no objection is made to the substitution of methylated spirits for rectified spirits in the preparation of soap, compound camphor, aconite, and belladonna liniments of the British Pharmacopœia.

2. No methylated spirits nor any derivative thereof, except sulfuric ether, ethyl chlorid, methyl chlorid, ethyl bromid, chloroform, and hydrate of chloral, can lawfully be present in any article whatever capable of being used either wholly or partially as a beverage, or internally as a medicine.

Generally it may be said that in addition to the alterations in the law in regard to the mineralized spirit, the old "ordinary" spirit is to be replaced by a new kind officially designated "industrial methylated spirits." In other words, the mixture of 9 parts of 90 per cent rectified spirits and 1 part of crude methyl alcohol is replaced by a mixture in which the proportion of wood alcohol or other approved denaturing substance is reduced to a minimum of one-nineteenth of the bulk of the spirit denatured. The result of this is that such denatured alcohol becomes suitable for the manufacture of the substances mentioned above, thus greatly extending the industrial uses of the denatured spirits.

The mineralized methylated spirit, sold for lamps and domestic use generally, contains, in addition to the above, three-eighths of 1 per cent of approved mineral naphtha. The following statement gives an idea of the production of denatured alcohol in Great Britain:

TABLE VI.—*Denatured alcohol produced in Great Britain, 1900–1904.*^a

Year.	Ordinary methylated spirit.	Mineralized methylated spirit.	Total.
	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>
1900.....	2,058,450	1,328,162	3,386,612
1901.....	2,075,514	1,439,243	3,514,757
1902.....	2,157,127	1,410,603	3,567,730
1903.....	2,213,580	1,464,672	3,678,252
1904.....	^b 2,139,784	1,527,573	3,667,357

^a Report of the English Industrial Alcohol Committee, 1905.

^b Decrease mainly due to the fact that certain firms were allowed to denature alcohol by other substances than wood naphtha.

FRANCE.

The general denaturing process followed in France consists in adding to 90 per cent alcohol 10 per cent of wood spirit (methyl alcohol), which contains 25 per cent of acetone and 2.5 per cent of various impurities. If the product is to be used for lighting or heating 0.5 per cent of heavy benzine is added in addition to the general denaturing agent, and when used for "finish" 4 per cent of gum resin is added. There are also special denaturing processes authorized for products or classes of products which can not make use of alcohol containing wood spirit. Examples of such cases are chloroform, for making which the alcohol is denatured by mixing with chlorid of lime in solution, and chloral, for which purpose a current of chlorin gas is past thru the alcohol. Not less than 440 gallons of spirit may be denatured by the general formula and not less than 220 gallons by any special formula.^a

The average price of denatured alcohol is difficult to establish, inasmuch as the 90 per cent alcohol which serves as a basis varies greatly in price. The denatured product does not sell for less than 11 cents a liter (1.05 quarts) at this time.^b It must also be remembered that the French Government has done much to encourage the growth of this industry. In June, 1906, a commission of analytical methods in regard to alcohol was named and prizes are offered for the improvement of the denaturing processes and for improved methods of utilizing alcohol for lighting. Since 1901 a bounty on alcohol manufactured in France and submitted to complete denaturation has been paid, which amounts to about 5 cents a gallon. The following table shows the growth in this industry during the past three years and the various purposes for which the denatured alcohol is employed:

TABLE VII.—Consumption of denatured alcohol in France, 1903–1905, inclusive.^c

Purpose for which used.	1905.	1904.	1903.
	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>
Heating and lighting.....	8, 328, 084	7, 654, 287	6, 922, 218
Varnishes.....	305, 222	328, 443	305, 909
Polishes, etc.....	46, 415	47, 552	66, 095
Plastic materials.....	562, 497	495, 874	530, 851
Manufacture of hats.....	6, 023	6, 287	9, 642
Dyeing and colors.....	14, 635	10, 329	14, 054
Rennet.....	3, 910	2, 932	3, 751
Collodion.....	11, 016	7, 185	3, 857
Chloroform.....	4, 914	4, 596	9, 959
Chloral.....	10, 091	7, 978	6, 499
Tanning materials.....	14, 978	40, 920	21, 081
Chemical and pharmaceutical products.....	178, 843	182, 410	300, 652
Scientific uses.....	31, 515	22, 824	13, 710
Ethers, fulminates, explosives.....	2, 961, 900	2, 375, 342	1, 687, 495
Total	12, 478, 043	11, 186, 959	9, 895, 773

^a Report of the English Industrial Alcohol Committee, 1905.^b Department of Commerce and Labor, Bureau of Manufactures, Daily Consular and Trade Reports, August 6, 1906.^c Sidersky, Bull. de l'Association des chimistes de sucrerie et de distillerie, November, 1905, p. 545; 1905 figures from Zts. Spiritusind., 1906, #9, No. 35, 323.

Beets, molasses, grain, and potatoes are the principal sources of industrial alcohol in France, the beets and molasses far exceeding other sources in importance.

GERMANY.

The general denaturing agent authorized in Germany consists of 1 part pyridin (obtained by distillation from coal tar) and 4 parts wood alcohol, to which may be added to each quart approximately 1.7 ounces of lavender oil or rosemary oil. In other words, the German denatured alcohol contains 2 per cent of wood alcohol and 0.5 per cent of pyridin, with optionally 0.125 per cent of lavender or rosemary oil.

Alcohol is completely denatured for use in motor cars, etc., by the use of 1.25 liters (1.32 quarts) of the general denaturing agent, 0.25 liter (0.26 quart) of a solution of methyl violet dye, and 2 to 20 liters (2.11 to 21.13 quarts) of benzol to every hectoliter (26.4 gallons) of alcohol.^a

There are many formulas for the incomplete denaturing of alcohol to be used for special purposes, of which the following are typical examples:

For industrial uses of all kinds: 5 liters (5.28 quarts) of wood alcohol or 0.5 liter (0.53 quart) of pyridin bases per hectoliter (26.4 gallons) of alcohol.

For various chemical preparations: 10 liters (2.64 gallons) of sulfuric ether, or 1 liter (1.06 quarts) of benzol, or 0.5 liter (0.53 quart) of turpentine, or 0.025 liter (0.026 quart) of animal oil, per 100 liters (26.4 gallons) of alcohol.

For soap making: 1 kilogram (2.2 pounds) of castor oil and 400 cubic centimeters (24.4 cubic inches) of soda solution.

For varnishes and polishes of all kinds: 2 liters (2.11 quarts) of wood spirit and 2 liters of petroleum benzin, or 0.5 liter (0.53 quart) of turpentine.

The following statistics regarding the production of spirits in Germany, the consumption of tax-free alcohol of different classes, and the materials used in its production, have been compiled by the Bureau of Statistics of the Department of Agriculture as showing the present development of the alcohol industry in Germany:^b

^a Report of the English Industrial Alcohol Committee, 1905.

^b Compiled from Vierteljahrshefte zur Statistik des Deutschen Reichs, 1906.

TABLE VIII.—*Production of spirits in Germany during the industrial years (October 1 to September 30) 1904-5 and 1903-4.*

Distilleries.	1904-1905.		1903-1904.	
	Distilleries in operation.	Alcohol produced.	Distilleries in operation.	Alcohol produced.
Agricultural distilleries:		<i>Gallons.</i>		<i>Gallons.</i>
Potato.....	6,048	75,860,933	6,059	80,304,589
Grain.....	7,620	8,615,719	8,934	7,593,514
Total.....	13,668	84,476,652	14,993	87,898,103
Industrial distilleries:				
Potato.....	21	149,864	22	151,158
Grain.....	780	11,612,490	788	10,699,810
Molasses.....	29	2,851,715	29	2,452,501
Other materials.....	39	4,253	39	2,695
Total.....	869	14,618,322	878	13,306,164
Other distilleries working up nonfarinaceous materials.....	57,635	958,145	50,160	614,750
Grand total.....	72,172	100,053,119	66,031	101,819,017

TABLE IX.—*Consumption of tax-free alcohol in the German Empire.*

Industrial year, October 1 to September 30.	Total tax-free alcohol.	Fully denatured.			Partially denatured.
		By the usual method.	By the admixture of benzol.	Total.	
	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>
1904.....	36,943,805	24,418,052	1,471,004	25,889,056	10,353,615
1903.....	36,760,600	24,604,952	1,393,867	25,998,819	10,195,535
1902.....	33,779,735	23,002,206	778,113	23,780,319	9,529,405
1901.....	29,324,191			18,616,826	9,137,482
1900.....	30,534,591			20,665,887	8,975,281
Industrial year, October 1 to September 30.	Without denaturation.				
	In public hospitals, etc.	In public scientific institutions.	In military technical institutions.	For making smokeless powder, fuses, and fulminates.	Total.
	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>
1904.....	74,206	53,679	10,752	562,497	701,134
1903.....	65,937	50,905	3,540	454,954	575,336
1902.....	58,672	51,566	15,903	343,870	470,011
1901.....	53,125	51,619		458,995	^a 1,569,883
1900.....	48,634	46,890		457,569	^b 893,423

^a Of which 1,006,144 gallons were used in the manufacture of medicines.^b Of which 340,330 gallons were used in the manufacture of medicines.

TABLE X.—*Materials worked up in distilleries in Germany during the industrial years (October 1 to September 30) 1904-5 and 1903-4.*

Materials.	1904-1905.	1903-1904.
	<i>Tons.</i>	<i>Tons.</i>
Potatoes.....	2,734,443	2,900,461
Rye.....	126,416	119,510
Barley.....	186,359	184,323
Corn and dall.....	121,350	93,319
All other grains and farinaceous stuffs.....	22,728	21,556
Molasses, beets, and beet juice.....	45,497	40,027
Other materials.....	1,182	1,143
Total.....	3,237,975	3,360,399
	<i>Gallons.</i>	<i>Gallons.</i>
Brewery refuse and yeast broth.....	2,123,742	2,273,474
Seed fruit and pomace.....	7,167,540	2,317,880
Stone fruit.....	10,359,400	5,049,372
Fruit and grape wine.....	871,497	554,651
Wine lees and reaps.....	16,359,414	14,109,293
Other materials.....	3,004,881	2,274,847
Total.....	39,886,474	26,579,517

As is shown in Table X, potatoes are by far the most important source of industrial alcohol in Germany where this crop is cultivated with a special view to its alcohol-producing qualities. The retail price of denatured alcohol (95 per cent by volume) is 29.69 cents a gallon, 90 per cent alcohol costing 27 cents, while gasoline sells for 20.8 cents a gallon and kerosene at 15 cents a gallon.

In comparing the figures for Germany with those for the United States it must be remembered that the German alcohol is 95 per cent commercial strength, and hence the number of gallons given must be almost doubled to compare with the taxable gallons of 50 per cent strength produced in the United States. The total number of taxable gallons produced in the United States during 1905, other than from fruit, was 147,810,794, and from fruit, 5,448,584, making a total of 153,259,378. The figures for Germany show a total of 100,053,119 gallons, or about 200,000,000 United States taxable gallons. Thus the German output, in round numbers, is 50,000,000 taxable gallons greater than that of the United States. With the vast agricultural resources of this country there will be no difficulty in increasing the consumption of industrial alcohol in the United States to equal or exceed that of Germany. This increase, however, must come slowly and probably after many disappointments, both of an agricultural and a technical character.

2
Issued December 9, 1912.

U. S. DEPARTMENT OF AGRICULTURE.

FARMERS' BULLETIN 516.

THE PRODUCTION OF MAPLE SIRUP AND SUGAR.

BY

A. HUGH BRYAN,

Chief Sugar Laboratory, Bureau of Chemistry,

AND

WILLIAM F. HUBBARD,

Forest Assistant, Forest Service.



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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., August 15, 1912.

SIR: I have the honor to transmit herewith a manuscript entitled "The Production of Maple Sirup and Sugar," by A. Hugh Bryan, chief of the sugar laboratory, Bureau of Chemistry, and William F. Hubbard, of the Forest Service, and to recommend its publication as a Farmers' Bulletin. The part by Mr. Bryan is the result of extensive chemical study of pure maple sirup and maple sugar, which included inspections of all the sugar maple districts of the United States. Mr. Hubbard's contribution includes the descriptions of the various sugar maples and the forester's recommendations for the management and care of sugar maple groves.

The manuscript as a whole contains the essential parts of Forest Service Bulletin 59 and Farmers' Bulletin 252, which, through the cooperation of the Forest Service, it is designed to supersede.

Respectfully,

R. E. DOOLITTLE,
Acting Chief.

HON. JAMES WILSON,
Secretary of Agriculture.

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Division of Forestry
University of California

THE PRODUCTION OF MAPLE SIRUP AND SUGAR.

INTRODUCTION.

The production of maple sirup and maple sugar is purely an American industry, Canada being the only country outside of the United States where they are made. At the time of the Napoleonic wars sugar was produced from the maple trees in Bohemia, and the industry received substantial means of encouragement from the Government of that country. Large groves of maple trees were planted and much attention paid to them, but, as the yield of sugar was small, and such a length of time elapsed before the trees could be tapped, the industry soon died out. At about that time the sugar beet came into prominence.

The earliest explorers in this country found the Indians making sugar from the maple tree, and in some sections, especially along the St. Lawrence River, producing it in quantity for trade. The white people began to make maple products and to apply more advanced methods to their manufacture. The crude methods of the Indians were soon improved upon, but beyond the tapping and boiling the general process remains the same as at that time. For many years, especially with the early settlers of the northern part of the United States, and even down in Kentucky and Virginia, maple sugar was the only sugar used. In the reports of the early meetings of the numerous agricultural societies are letters from various sugar makers describing their attempts to produce an article equal to the imported sugar, or muscovado, of the West Indies. Cane-sugar methods of manufacture were tried, with varying degrees of success. A few refineries for producing white sugar were built and operated with maple sugar as their raw supply. The iron kettle, birch-bark tank, wooden spiles, and old way of tapping yielded a dark, ill-tasting product, but with care and changes in methods and apparatus the products were improved. In those days many trees were killed by the crude methods of tapping, and much stress was laid upon this operation by the early writers.

To-day maple sugar and maple sirup are considered delicacies and are not produced in such quantities as formerly. The aggregate of all pure maple products and the many imitations may be the same, for it is said that were all the maple trees cut down, and thus the supply of maple sap cut off, no drop in the quantity of sirup or sugar would be noted. Within the last six or seven years there has been a renewed tendency toward the production of a better grade of maple products.

Maple sirup or maple sugar prepared by the best methods is a light-colored, pleasant-tasting product. Contrary characteristics are the result of uncleanly methods and possibly sour sap in ninety-nine cases out of a hundred, and were manufacturers to take greater care in their methods their results would show improvement. This better grade may not appeal to many people who have been accustomed to the dark, stronger grades.

In visits to the many maple camps for the collection of samples of pure maple sirup and sugars for analysis, much attention was paid to the manufacturing side. The results of the work on maple-sap sirup are given in Bulletin 134 of the Bureau of Chemistry; the work on maple sugar and maple-sugar sirup has not been completed. As these articles are to a great extent technical, it was thought the work should be compiled for the layman or the small sugar maker.

SUGAR MAPLES.

All the maples have sweet sap, but only from a few of our native species has sugar been made in paying quantities. The first place is held by the sugar maple (*Acer saccharum*) and a variety of it—the black maple (*Acer saccharum nigrum*). The red maple (*Acer rubrum*), the silver maple (*Acer saccharinum*), and the Oregon maple (*Acer macrophyllum*) are of less importance, and the box elder (*Acer negundo*) is least important of all.

THE SUGAR MAPLE.

The sugar maple spreads over a wide area, but as a tree for the production of sugar in paying quantities its range is limited to eastern New England, New York, Pennsylvania, the southern Appalachians, the Ohio Valley, and the Lake States and adjacent parts of Canada. In the Gulf States and as far north as southern Arkansas the tree is represented by a variety (*Acer saccharum floridanum*) from which no sugar is made.

The sugar maple is a stately and vigorous forest tree, capable of growing in dense stands. It bears a plentiful crop of seeds, which in most localities ripen in the early fall. These seeds germinate readily, and under favorable circumstances the entire forest floor is heavily carpeted with seedlings, the succulent, sweet foliage of which is eagerly devoured by all kinds of stock. The young seedlings are very thrifty and can stand the shade of a complete forest cover. This tolerance of shade is one of the distinguishing features of the sugar maple, and, although it is less pronounced in later years, the mature tree has one of the most persistently heavy crowns in the forest.

Seedlings, although not killed by complete shade, are kept suppressed and grow slowly; but if they have germinated in the open, or the forest above them is removed, they grow up into thickets of remarkable density. In such a condition the struggle between the young trees is so fierce that the development of even the most thrifty is seriously retarded. The species being so tolerant of shade and by nature so vigorous, no individual gives up the struggle, but does its utmost to overtop the others and gain the sunlight. As a result

the stand keeps its extreme density for a long period, and each tree grows long and spindling. The forest-grown tree develops slowly on this account, and has a long clean stem and a small crown, while the roadside maple has a short trunk and a great egg-shaped crown of dense foliage. The root system tends to be shallow, with many laterals and an undeveloped taproot. In the forest this character is more marked than in field or roadside specimens, and any sudden opening up of the stand may result in loss by windfall or by a drying out of the roots.

There is no doubt that the quantity of sap that a tree yields stands in direct relation to the size of its crown, but many sugar makers believe that trees in a forest produce more sap than those in a grove. The explanation is found in the fact that the forest floor with its covering of litter and humus contributes to the vitality of the trees more than the grass carpet of a grove. To obtain a heavy sap production a complete crown cover and a rich deposit of humus are of vital importance.

Within its wide range the sugar maple appears as a predominant tree only in the New England States, New York, southern Canada, northern and western Pennsylvania, and in parts of Ohio, Indiana, Illinois, Michigan, Wisconsin, and Minnesota. In the southern Appalachians it occurs in scattered bodies where climatic conditions are similar to those of the North, confining itself chiefly to north slopes or to the coves, on moist, well-drained, rich soils where the heat of the sun is tempered. As a rule it associates with the beech, birches, and basswood, but also mixes with the yellow poplar, hickories, and other hardwoods, and with hemlock and some of the eastern spruces. At the heads of the coves and in the bottoms it often forms pure stands fit for commercial tapping, and its reproduction is everywhere excellent.

In its northern home it is a principal forest tree and often forms from 25 to 75 per cent of the total stand. It prefers a moist but well-drained soil, and seems to do its best on glacial drift or on rocky hillsides and benches. In the cool atmosphere of this region all aspects are equally acceptable, but it avoids or grows sparsely on ridge crests, generally leaving the ground in such situations to spruce or to beech. In the extreme northern part of New York State and the adjacent district of Quebec the forest growth is often almost pure maple, and even considerably farther south, where beech and birch become associate trees, the sugar maple holds its own as the dominant species. In northern Pennsylvania, at an altitude of over 1,000 feet, the mixture is much the same as it is in New York. In Ohio, Indiana, and Illinois a similar condition is found; but as the hilly country disappears the maple retreats to the richer and damper soils, leaving great areas to the oaks, chestnuts, etc. This is particularly true of the southern parts of these States. By the time the western and southwestern limits of its range have been reached it has only a scattered occurrence, even in the most favorable positions. In southern Michigan the forests are similar to those in New York, but as one approaches the pine region of the North the maple confines itself to the more fertile places. The same is true in Wisconsin and Minnesota, where the sugar maple reaches its northwestern limit in the United States.

Maple trees are often seriously injured by an insect commonly called the "maple worm," concerning which information may be had from Circular 110 of the Bureau of Entomology, United States Department of Agriculture.

In the present discussion the sugar maple is not considered as a lumber tree, for which a long stem free of branches is desired, but rather as a paying producer of sap. Under this aspect a silvicultural problem is presented radically different from that which ordinarily confronts the forester. (See p. 9.) The full and heavy crown with a large leaf surface must be developed in place of the long, clear stem. The sap flow must be continuous and plentiful. The best sap flow is where the transition from winter to spring is slow, where the days are warm and sunny and the nights frosty. These conditions do not occur throughout the entire range of the species. A locality wherein the ground thaws quickly and which has no great variation of temperature between day and night is not suitable for sap production. The "season" must be long enough, also, to insure sap in merchantable quantities. Such conditions are characteristic only in the Northern States, and as sugar making extends farther south it can be profitable only at altitudes which reproduce the climatic conditions of the North.

THE BLACK MAPLE.

The black maple is generally considered superior to all others as a producer of sap. How far this is true is uncertain. In its general silvical characteristics it is similar to the sugar maple, save in the fact that it seems to prefer lower land, such as the banks of streams and rich alluvial river bottoms. It is found in Vermont on the shores of Lake Champlain, and ranges southward, west of the Alleghenies, from Minnesota to Arkansas and eastern Kansas.

THE RED MAPLE.

The red maple has the widest range of all its family in America. The natural home of this tree is along the borders of streams and on low, swampy ground. In the North it often forms a pure growth in such places, but it is along the Ohio and the Mississippi and their tributaries that it reaches its greatest perfection. Like the sugar maple it is tolerant of shade, and seedlings sprout plentifully from the heavy crops of seeds, which ripen in the late spring or early summer. As a swamp tree it associates in the Southern and Middle States with the sweet magnolia and loblolly bay, the bald cypress, various oaks, and the red, black, and cotton gums. It does well, also, on less moist lands. It is generally of vigorous growth, but the grown trees are inclined to unsoundness at the butt. As a sugar-producing tree it enters into consideration in the Middle and Western States only where the sugar maple is not plentiful. It has an abundant flow of sap which is much lower in sugar content than the sugar maple. On account of its early flow of sap it is often tapped at the first of the season to produce the earliest maple products.

THE SILVER MAPLE.

The silver maple ranges from New Brunswick to western Florida, and west through southern Ontario and Michigan to eastern North and South Dakota, Kansas, and Indian Territory. In the North it appears in mixture with the sugar maple, but in general prefers lower altitudes and moister soils. It reaches its greatest perfection in the valleys of the Ohio and Mississippi, where it is one of the characteristic trees on the lowlands of these rivers and their tributaries. The flow of sap is plentiful and sweet, but, like that of the red maple, liable to discoloration, and the season is short and uncertain. It is, like the red maple, only to be considered as a sugar tree outside the region where the sugar maple is a dominant species.

THE OREGON MAPLE.

The Oregon maple is the only western species which can be considered as a producer of sugar. In localities where the season is favorable the sap is of good quality and the flow considerable. The tree is found west of the Cascades and Sierra Nevada, from the Canadian border to southern California. It prefers rich, moist soil, and reaches its best development in the river bottoms of Washington and Oregon. The census of 1910 reports a very small production (10 gallons of sirup) from Columbia County, Wash.

SUGAR GROVES.

GENERAL CONSIDERATIONS.

The ideal sugar grove should contain that number of trees which will give a maximum yield of sap per acre; whence it follows that the formation of a grove must consider the yield per given area rather than the yield per tree. To determine the exact number of trees that should occupy an area would take many years of experiment, but directly and indirectly there has been much information collected on the subject of sap production through a study of individual trees, and from this a number of safe deductions can be made. An equal amount of sunlight being given, the sap and sugar production is proportionate to the leaf area of the tree. This statement is corroborated in a bulletin of the Vermont Agricultural Experiment Station,¹ where it is also asserted that the sugar production of the tree depends more on the actual leaf area than on the amount of light which it receives. In other words, if a small-crowned mature tree be set free to light on all sides, the sap production will be stimulated only to a very slight extent. From this it follows that the number of trees per acre must be consistent with the greatest possible crown development of each tree in the grove. At the same time it is not to be forgotten that the maple is inherently a forest species. The large crown of foliage has an extensive leaf area for evaporation, and demands a protected soil which can keep it well supplied with water. Such soil is best found in the forest, where

¹ Vermont Agr. Exper. Sta. Bul. 103, December, 1903, pp. 117, 118.

the ground is kept heavily matted with leaves and humus, so that the sun and drying winds will have little access to it, and a comparatively uniform degree of moisture and coolness may be maintained under all conditions. Commercial sugar making is confined to a small part of the botanical distribution of the sugar maple, because of a peculiar climatic requirement. It is the gradual northern spring, with the slow yielding of the frost by the ground, which makes the sap flow long and continuously enough to give a paying production of sugar. A sudden thaw affects both the quality and the duration of the sap flow. On this account it is always desirable to maintain forest conditions in a sugar grove, for if the ground has a heavy carpet of leaves and humus, it will be less sensitive to changes in temperature.

Altitude is one of the most important factors in determining the necessary density of the sugar grove. High up in the mountains, where the summer is moist and the spring long, and in the North, the necessity of an unbroken cover is not so great as where the summer is hotter and the spring less gradual in its transition from winter to warm weather. In mountainous regions the forest can be more open, and in every large grove a section on a southern exposure will insure an early sugar season. In lower altitudes the close grove of full-crowned trees will have an advantage over a scattered stand of field trees exposed to the effects of a variable spring. It should not be forgotten, however, that trees which have developed from their youth in very open groves have stronger root systems than forest trees, and that they draw their water supply from the moist subsoil (see p. 7); but such groves have a relatively limited production per acre, and, while serviceable for a small home production, would cover too large an area to be profitable for a large undertaking.

The model grove should satisfy the following requirements as far as possible.

(1) It should contain the greatest number of trees per acre consistent with fully developed crowns.

(2) The forest cover should be unbroken, so that in summer little sunlight falls upon the ground.

(3) There should be a complete litter of humus and leaves, to the exclusion of grass and light-demanding weeds.¹

(4) Young trees should be kept in reserve to take the place of those that fail, and to fill other openings in the cover.

(5) No grazing should be allowed in the grove, except in special cases where the cover is perfect and no reproduction is needed. Cattle not only keep back all reproduction, but also do harm by trampling and breaking the ground, so that it dries out.

(6) The grove should be made accessible by a system of roadways to facilitate the collecting of sap. If the network is complete no difficulty will be found with the underbrush, which hinders sap gathering little in the early spring when the woods are devoid of foliage.

The first three points vary in importance with the latitude and altitude of the grove, but they are always worthy of consideration.

¹ French and German experiments have demonstrated that a heavy growth of grass dries the soil, and interferes with the entrance of water even during a heavy rain.

In discussing the methods required to bring about these results, the several common types of sugar groves will be described. Logically it would be proper to begin with the treatment of a sapling thicket and continue through each stage to the mature grove; but as the earlier stages of growth are the most complicated to deal with, the order of consideration will be reversed.

THE IMPROVEMENT OF A DENSE MATURE GROVE.

A large number of groves are merely parts of the old hardwood forest, having a preponderance of sugar maple in the mixture. These trees have their normal forest form—a long, smooth stem and compact crown. There is little to be gained in actual sap production by thinning such a stand (p. 9), as it has generally passed the period of vigorous growth and would not develop larger crowns, although the sap season may be brought on earlier by opening up the grove to sunshine. The mixture can be regulated, however, and provision made for a pure growth of maple to succeed the old forest as it passes away. The usual mixture of birch, beech, elm, basswood, and ash may be gradually removed and the reproduction of maple thereby assured. This thinning should pay for itself in most localities from the resulting fuel and saw timber. In making such a thinning the following precautions should be observed:

(1) When the trees to be removed occur in groups, they should not all be cut out immediately, leaving large gaps in the forest cover, since forest-grown sugar maples have a broad, shallow root system, and are subject to windfall when suddenly exposed. The trees which crowd the best maples should be taken out first; the rest should be removed later, when the sugar trees have become more wind firm.

(2) Where several maples crowd each other and form a dense cover, those with the smallest crowns, those which are unsound, and those which show signs of bad health or decline should be removed.

(3) Young maples which show possibilities of good crown development should be cut free from interference on every side.

(4) If the grove borders on open land, it should not be thinned for a distance of at least 25 feet from its edge. This is a safeguard against damage by storms, and is particularly important in borders exposed to heavy winds.

(5) When practicable, the young growth of other species than maple should be removed.

(6) It is well to accomplish the thinning in a series of years, rather than at once and radically, thus avoiding violent changes.

(7) It is important to maintain the humus and ground moisture in every maple grove. In localities where natural forests of sugar maple are common the danger of destroying the proper soil conditions by letting in the sunlight is not great, but if a grove of this type lies where the summers are hot the cover must be broken very gradually.

THE IMPROVEMENT OF AN OPEN MATURE GROVE.

In the more settled and less wooded portions of the maple sugar producing district it is noticeable that a large proportion of the groves are old and very often overmature. They have evidently

been left on favorable situations from the original forest, and as a rule no attempt has been made to renew them or keep up their vigor since the adjoining land was first cleared. A young and thrifty set of trees is a rarity among the great number of old, open, and grass-grown groves.

As a rule these groves are on small farms, where they are used quite as much for pasture as they are for sugar making. In cases where the pasturage can not be spared, and where sugar is only a small item in the farm production, there is little to be done for their improvement. When the grazing can be spared, however, and the owner desires to increase the sugar-producing capacity of his trees, it is undoubtedly better to bring about a reproduction from the old trees than by planting a new stand.

The first step to be taken in such a process of improvement in a more or less open and grass-grown grove is the exclusion of stock. After laying out proper driveways for sap gathering, the seedlings should be allowed to come up everywhere else. All unsound and dying trees should be cut out and young growth of all other species than maple removed. In a very short time the young maple seedlings will take possession of the open ground and grow vigorously where they get sufficient light. When they are 8 or 10 years old and 6 to 8 feet high, or more, the struggle for supremacy among them will begin. In each opening large enough to permit the development of a tree with a full crown, the strongest and most thrifty seedling which has a favorable position should be selected, and the heads of those within a radius of 12 feet or more about it lopped off with a corn knife. The crowns of at least two-thirds of these trees must be removed; the remaining crowns will insure a good ground protection and leaf fall until the favored tree has filled the opening. In the case of small openings the thicket should remain unthinned; the struggle between the trees will keep them all suppressed, while they will supply the necessary ground cover. The seedlings which come up under the direct shade of the old trees will never develop to any size, unless some of the large trees are removed by age or accident. Cattle may be let in the grove when it has become too tangled for convenient sap collecting and when the young growth desired for open places has reached a height of 8 or 10 feet. They will soon open up the smaller and undesirable growth. At the same time roadways should be opened and the ground kept free of fallen limbs and trees. The tall, slender seedlings will be a small obstruction in sap gathering, but a little discomfort can be borne for the sake of the undoubted advantages obtained by a ground cover.

THE IMPROVEMENT OF A DENSE YOUNG GROVE.

In many parts of the maple-producing section a second-growth forest has come up similar in composition to the original stand. The sugar maple often forms a predominant part of such a wood, and in that case all that is needed to turn it into a sugar grove is to remove a number of interfering trees, thus giving the proper number of maples a chance to develop the full crowns necessary to a maximum yield of sap per acre. Preference should be given to the younger

and more thrifty stands, where the trees are just entering the period of most vigorous development.

The difference between thinning a young stand and a fully matured grove of the same type is usually that in the former case provision must be made for growing a set of full-crowned sugar trees from the more thrifty of the young maples. In a stand from 40 to 60 years old it is easy to pick the largest and best-developed specimens and favor them for the future. Some of the directions to be observed in treating a dense young grove are the same as those given for the treatment of the mature grove.

(1) Select the sound, dominant trees which show a natural tendency to a well-branched, compact crown of large size, and remove from all sides everything which tends to crowd them. If the stand is between 40 and 60 years old, leave about 100 trees to the acre; if older, leave about 75 trees. The average healthy young maple can be freed for 10 to 12 feet on all sides of its crown without the slightest danger, except in the most exposed positions.

(2) In the choice of sugar trees the position and influence of each on its neighbors must be considered. If two dominant trees crowd each other seriously, remove the least promising.

(3) In case the beech, birch, or other species are so grouped that their removal would make a serious gap in the forest, it will be well to let several of them stand, but they should be so treated that maple seedlings (which nearly always gain possession of the soil even under beech) will have light enough to come in under them. When these seedlings become established the beech or birch can be removed, and young maples favored. When practicable always cut out other seedlings than maple.

(4) Successive thinnings are better than a radical opening up of the stand, because in this way danger of windfall and drying out the soil are avoided. This method also leaves room to overcome the damage done by porcupines. These animals probably are the worst enemies of the young maple. One porcupine in a single night can strip the bark off many saplings, and to such an extent that they are permanently ruined. The sugar trees should not have more than 10 or 12 feet of free space on any side of their crowns. A thrifty maple can fill such a gap in eight or ten years, after which a final thinning may be made and the remaining weed trees removed.

(5) The edges of the grove which border upon open land should not be thinned enough to leave the stand unprotected from strong winds and sunlight. If other species are crowding the dominant maples, they should be removed; but, as a rule, the borders should remain dense and the trees should be covered to the ground with foliage.

(6) In cool situations, or in elevated regions, the thinning may be heavier than farther south or in lower lands, where more care is necessary to preserve a proper ground cover. Firewood and other timber secured by thinning should pay for the cost of the operation. The necessity for well-located roadways to take out the sap should not be forgotten.

THE MANAGEMENT OF A SAPLING THICKET.

Throughout the maple region dense thickets of young saplings are common in abandoned fields and pastures. Where a sugar grove is desired, it will pay to take such young growth in hand if no old trees are available in sufficient numbers. Left to themselves, the young trees usually become so densely crowded that even when 20 feet high they number from 2,000 to 3,000 to the acre. Under such conditions growth almost ceases even in the dominant trees, and at a time which, in normal stands, is the period of most vigorous growth.

The first thinning should be made when the saplings are about 6 or 8 feet high, if the owner feels justified in helping them at this time. The largest and healthiest trees, on an average about 12 feet apart, should be selected, and the tops of the others cut back with a hatchet or a corn knife in such a way that they can not overtake the favored individuals.

Cutting back trees in this manner can be done very rapidly. Three men should cut over an acre a day. Although there is no return in firewood or other material from such early thinnings, the young trees will be given a favorable start in their development at the most critical period of growth. The ground cover at the same time will be kept intact by the sprouts, until the selected trees fill out and close up the space with their crowns. When they are about 25 years old the dominant trees, which are about 12 feet apart, will begin to crowd each other, and another thinning must be made to give the best ones room. Experiments are under way to show how much time will be gained by this method in obtaining a stand fit for tapping. All general forest practice shows that the gain over the unthinned thicket should be at least 25 per cent.

If the thicket to be turned into a sugar grove contains older and larger trees than have been considered, a regular course of thinning should be instituted. The main points to keep in mind in this case are as follows:

- (1) Choose the thrifty trees which show a tendency to good, symmetrical crown development, and set their crowns free on all sides to a distance of about 12 feet. See that the selected trees are sound and free from forks which may develop badly.

- (2) Remove all long, spindling trees which are likely to bend over.

- (3) For ground cover, leave all specimens which do not threaten the crowns of the chosen trees and which are capable of casting a small amount of shade.

- (4) Remove all species but maple, except when they are very much suppressed. Low, broad-crowned trees of any kind will help to shade the ground.

- (5) Do not disturb the borders of a dense thicket. Sun and wind must be excluded from a stand which has been suddenly opened up within, and which is unaccustomed to the new conditions.

To make this system of treatment clearer, a concrete case will be described. In the autumn of 1903 a stand of young maples in Vermont was thinned by a member of the Forest Service. The stand is situated at an altitude of about 1,200 feet, with a southeastern exposure. It came up in an abandoned meadow, which was seeded

from a few old trees along a bordering wall. The dominant trees are from 30 to 40 feet in height and from 15 to 25 years old, with an average diameter for the stand, suppressed trees included, of 2 inches, breasthigh. The entire tract is very dense, and, although the extreme difference in the age of the trees is about 10 years, the difference in their size is far greater than the discrepancy in age would explain. More than half the stand is 1 inch and under in diameter, and yet



FIG. 1.—Stand of maple saplings in need of thinning.

many of these trees are as old as near neighbors three times as large. This results from the extreme vitality of the sugar maple, and shows the urgent necessity for thinning at an early age. Two plats, each 0.7 acre in size, were thinned, with the following results:

Thinning of a maple-sugar thicket, showing the number of trees per acre of various diameters in the original stand, the number removed, and number left.

Diameter breasthigh.	Number of trees per acre.		
	Original stand.	Removed.	Left.
<i>Inches.</i>			
0.5 to 2.....	1,517	755	762
2 to 4.....	1,042	653	389
4 to 7.....	309	126	183
Total.....	2,868	1,534	1,334

Figures 1 and 2 show the tract before and after thinning. Twelve cords to the acre of fair firewood were cut, an amount which should

ordinarily pay for the thinning. The large number of small trees left after thinning is noticeable in the illustration, and is a point not to be overlooked. All trees that in no way interfered with the dominant stand and had a fairly full crown were allowed to remain as cover. There is no chance of their overtaking the favored trees, and they furnish the needful shade whereby a more radical opening of the crowns in the dominant stand is permitted. The final trees of the grove are to be selected from the trees which are 4 to 7 inches in diameter, the remainder acting as a reserve in case the selected trees should meet with accident. The heaviest cutting was made in that part of the stand which ran from 2 to 4 inches in diameter, the class which interfered most with the future sugar trees. Those individuals which gave promise of becoming members of the final



FIG. 2.—The same stand shown in figure 1 after thinning.

stand were given more room than the others. Although the cutting took away such a large proportion of the stand, it will be observed that the trees are still in close order. This will necessitate a later thinning, probably after about six years, but at present further thinning would subject the long, slender saplings to danger of overthrow and the ground to drying.

SITUATION OF A SUGAR GROVE.

The best location for a sugar grove is where the maple thrives best under natural conditions. In the Appalachian region this will be in the north coves, and in Ohio, Indiana, and adjacent States on rich, moist, gravelly soils. In the Northern States, where the maple flour-

ishes on all exposures, the exposures to the south are generally to be preferred, because there the sap runs earlier, and the first sirup and sugar to reach the market obtain the best prices. On northern exposures and in very dense forests the sap season begins later; but if the sugar grove is to be on a large scale, it will be well to have it include both southern and northern exposures, so that the run of sap may be continued longer and not come at once in a quantity too great to be easily cared for. In the Northern States the best sugar groves are usually on rocky slopes with soils rich in humus, at an altitude of about 1,000 feet.

PLANTING A SUGAR GROVE.

The advisability of planting a sugar grove will depend partly on the locality. The problem presented is notably different in the Middle West and in the region of commercial production in the Lake States and the Northeast. In the West maple-sugar production has steadily declined and shows no sign of a revival. The planting of sugar groves in this region is, therefore, not generally advisable.

In the region of commercial production it is usually easy to find old groves, young stands of second growth, or sapling thickets which can be made productive more quickly than a plantation of seedlings. In cases where no such beginning is possible, and a plantation has been determined upon, the following suggestions may be useful:

Avoid planting the trees too far apart. This is the mistake most commonly made. Wide spacing deprives the soil of its needful protection, reduces the yield of sap per acre, and gives a poor return for the expense of planting and for the amount of land used. Planting should always be done in early spring; and as the regions in which it is likely to be necessary are usually at low altitudes (see p. 7), it will be good policy to plant the trees close enough to insure a proper ground condition from the first. This will be best accomplished by setting the trees 6 by 6 feet apart. This gives 1,210 trees to the acre, which will not prove very expensive, as small seedlings, costing about \$2 per thousand, may be used, or they may be gathered from the woods, preferably in wet weather. When this is done, care should be taken to select thrifty specimens, not over 2 feet in height, and to plant them immediately.

When the young trees reach a height of about 10 feet and begin to crowd one another, the grove should be treated in the same manner as that recommended for the wild sapling thicket (see p. 14). This will give a maximum number of full-crowned trees to the acre, and the proper ground conditions will be maintained.

In most cases it will be well to cultivate the ground for one season, or possibly two, but the soil should acquire the forest character as soon as possible. Where that is not readily attainable, a maple grove is not likely to pay.

In some situations it may be advisable to mix with the maple a number of quick-growing trees valuable for posts or farm lumber, in order to secure early returns on the investment. The best species to use in this way can be determined only for definite localities. Advice in such cases will be furnished willingly by the Forest Service.

THE PREPARATION FOR OPENING.

In the spring, when preparing for the opening of the sugar season, or during the fall of the year before, roads should be cut through the bush. Any brush that is in the way should be cut down, and any holes that might overturn the cart of sap should be filled. In laying out the roads through the woods some plan should be followed that every portion of the bush may be accessible, so that when the sap is running fast and many sap buckets quickly fill the gathering pail, the latter will not have to be carried far to be emptied into the hauling tank. Furthermore, by having these roads accessible to all parts of the bush there is more assurance that all the sap buckets will be visited each day during the sugar season. The necessity for this will be discussed later.

TAPPING THE TREES.

LOCATION AND CHARACTER OF TAP HOLE.

Before tapping, the side of the tree should be brushed with a stiff broom to remove all loose bark and dirt and a spot selected where the bark looks healthy, some distance from the scar of a previous tapping. Care should also be taken to tap where a bucket attached to the spout inserted in the hole will hang level and be partly supported by the tree itself. The distance from the ground should be such as will be convenient for the collector of sap; that is, about waist high. The location of the tap hole as to the point of the compass depends much upon the tree and its location in the bush. In general it is on the south side of a tree for earliest runs, as the sun shines on this side first, the east side being next best, while it is claimed by some that holes on the north side flow longest. It is usually best to tap on the side of the tree where other trees do not shade the spot.

The main requisite in tapping a tree is a good sharp bit with which a clean-cut hole can be made. If rusty and dull, the bit cuts a rough, feathered hole, which soon becomes foul, stopping the flow. After the tapping all shavings should be removed to make the hole clean. The bark should never be cut away before boring the hole, as this shortens the life of the tree.

SIZE OF HOLE.

Among sugar makers the size of the hole is a much mooted question. All agree that it should be of such size that it will heal over in one season, or at the longest in two years. General practice seems to indicate that three-eighths to half an inch is the best diameter; then, if the season is long and a warm spell interrupts the flow, the holes can be reamed out to one-half to five-eighths of an inch, and thereby secure an increased run. A thirteen thirty-seconds of an inch bit is often used. The bit should be especially sharp and should bring the shavings to the surface. Its direction is slightly upward into the tree (see fig. 3). The slant allows the hole to drain readily.

The depth of the hole is an important point on which different makers vary. It should, however, be regulated by the size of the

tree, as it is only the layers next to the bark that are alive and contain enough sap to flow freely. Toward the interior the flow diminishes. With the ordinary tree a hole not over $1\frac{1}{2}$ to 2 inches deep is best; some bore in 3 inches, but this is possibly too deep. In small second-growth trees a short incision, or just through the sapwood, is best. In any case boring should be stopped when dark-colored shavings appear, even though a depth of an inch has not been obtained, as this shows dead wood and that the sapwood has been passed through.

NUMBER OF TAP HOLES TO A TREE.

It is far better to make it a rule to tap only one place on a tree; by so doing the life of the tree is prolonged. Large first-growth trees might be tapped in two and sometimes three places without injury, but it is imprudent and disastrous to tap in two places near together so that the sap from the two is collected in one bucket.

TIME FOR TAPPING.

It is good policy to tap early in the season in order to obtain the earlier runs, which are generally the sweetest and therefore the best producers. Makers have lost half and even more of their crops many seasons by not being prepared for the first runs. It is hardly necessary to describe what constitutes "sugar weather," as all

sugar makers are familiar with the term. In general, it might be said that during the middle or last part of February in the southern sections and later in the northern ones, when the days are becoming warm, the temperature going above 32° F., and the nights are still frosty, the season is ready to open. If the days are very bright, warm, and sunny, the sap will start with a rush, but soon slacken, or if a high wind starts up the flow is checked. A protracted warm spell or a heavy freeze, with days and nights of even temperature, causes the flow to stop altogether, to start up again when the meteorological conditions are right.

Mr. Phillips, of Ohio, has kept the date of tapping and also the last day of boiling for his camp from the year 1880 to date. The data are as follows, showing how the opening and closing dates of different seasons may vary.

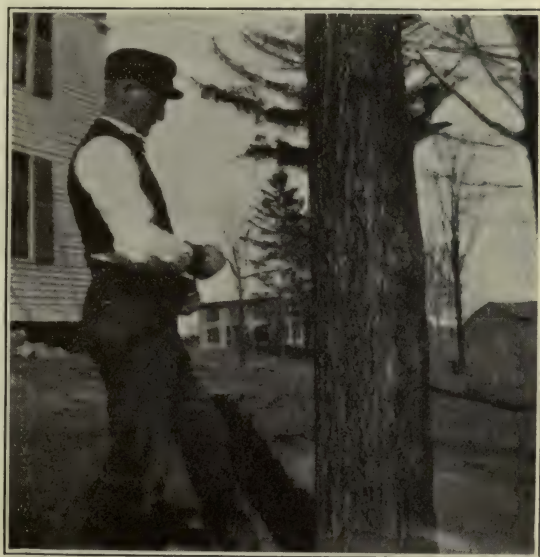


FIG. 3.—Tapping the tree.

Table showing date of opening and closing of a sugar camp in Ohio from 1880 to 1912.

Year.	Opening date.	Closing date.	Days.	Year.	Opening date.	Closing date.	Days.
1880	Feb. 24	Apr. 1	37	1897	Mar. 8	Mar. 31	23
1881	Mar. 9	Apr. 16	38	1898	Mar. 3	Apr. 11	39
1882	Mar. 2	Apr. 1	30	1899	Feb. 20	Apr. 11	50
1883	Mar. 1	Apr. 10	41	1900	Mar. 8	Apr. 14	37
1884	Mar. 12	Apr. 14	33	1901			
1885	Mar. 27	Apr. 18	22	1902	Mar. 7	Apr. 6	30
1886	Mar. 15	Apr. 11	27	1903	Feb. 26	Mar. 15	17
1887	Mar. 2	Apr. 9	38	1904	Mar. 2	Apr. 6	34
1888	Feb. 21	Apr. 10	50	1905	Mar. 16	Mar. 29	13
1889	Mar. 11	Apr. 9	29	1906	Feb. 13	Apr. 2	48
1890	Feb. 17	Apr. 7	49	1907	Mar. 14	Mar. 23	9
1891	Feb. 13	Apr. 11	57	1908	Mar. 5	Mar. 26	21
1892	Feb. 22	Mar. 30	37	1909			
1893	Mar. 7	Apr. 3	27	1910			
1894	Feb. 27	Apr. 7	39	1911	Feb. 16	Apr. 4	47
1895	Mar. 23	Apr. 12	20	1912	Mar. 17	Apr. 9	24
1896	Feb. 27	Apr. 10	43				

APPARATUS USED IN THE MAPLE INDUSTRY.

SPOUTS.

The spout, or spile, as it is often called, is the tube through which the sap flows into the bucket. It is usually of metal, but often hollow reeds are used. The best forms are perfectly cylindrical, smooth, and of an even taper, making them easy to insert and to remove without interfering with the wood tissue. The perfect spout should be strong enough to support the bucket of sap safely, and for obvious reasons should bring the whole weight on the bark of the tree and not on the inner tissue or sapwood. Spouts with spurs or anchors are to be avoided, as they tend to split the bark and crush the sapwood, thereby decreasing the flow and allowing the sap to leak. The spout having a small hole is best, because one with a large hole allows the bore to dry out faster when there are strong winds. There are various forms of spouts on the market (see fig. 4) that meet these requirements, and there are also forms that can be made at home that will serve the purpose. A spout should have a hook or stop on which the bucket is to hang, unless the bucket may hang on the spout itself. It is bad policy to drive a nail below in the tree for this purpose.

Many makers use wooden reeds as spouts. These as a rule are not strong enough to hold the bucket and, moreover, soon become foul from bacteria and souring of the sap. Their use generally leads to a decreased yield and to the production of a poor-grade sirup.

Great care should be exercised in driving the spouts into the tree not to compress the sapwood or split the bark. Both conditions prevent a good yield of sap and the latter tends to a decay of the tree or a bad healing of the hole. The same care should be used at the end of the season in removing them. Perfectly round spouts with the proper taper should be easily removed by turning without injury to the tree.

SAP BUCKETS.

Buckets are suspended on the tree to catch the sap. Formerly hollowed logs or birch-bark boxes were the usual forms, and these



FIG. 4.—Common forms of sap spouts. (Nos. 1 and 12 are wooden; nos. 3, 4, 7, 10, 13, 16, 17, and 18 are molded metal; the others are metal bent into shape usually over forms.)

are found to-day in some of the cruder camps. Their use does not lead to the better grades of maple products, and because they are

generally placed on the ground and not suspended to the tree they cause a considerable loss of sap.

Wooden buckets were formerly used almost universally and are still used quite extensively. As a rule they are heavy and do not hang well on spouts or they require a nail to be driven in the tree below the spout to hang on. The latter practice has been mentioned as inadvisable. Wooden buckets are a great care to the maker to keep the hoops tight to prevent their falling to pieces or causing loss of the sap from their leaking. Other points against their use are the large space they occupy in storage between seasons and the difficulty of keeping them clean; the sap enters the wood and, besides extracting coloring matters and flavors, they soon become sour when the warm days come. To prevent these conditions makers who use this form of buckets paint them on the outside, and in many cases on the inside, during the summer or fall. (The kinds of paint used are taken up later.) By this means the pores of the wood are filled and the wood preserved from decay. Most makers are replacing their wooden buckets as they become useless with some form of metal buckets, although there are a few who retain them, believing they impart a pleasing flavor to the sirup.

The most satisfactory buckets are of metal, practically free from corrosion or rust, and fitting well to the tree. They are light in weight, yet strong, not easily dented, are readily cleaned and easily stacked away in a small space after the season closes. As to the metal composing the bucket, the finest, no doubt, would be aluminum, but the expense is too great. The heavy tin plate comes next, with the lighter tin plate coming in order, then the galvanized-iron buckets. One form that is certainly objectionable is the "terne-plate" bucket. "Terne plate" is iron coated with a mixture of tin and lead in about equal parts. The objection to the lead is readily understood. For the same reason there is objection to a bucket that is soldered on the inside. The seams should be turned, and, if soldered, this should be on the outside, so that the sap does not come in contact with it. Objection has also been raised to galvanized buckets in that they soon begin to peel, leaving the iron surface to rust.

The widely flaring bucket, such as the ordinary tin water bucket, is not a good form. One with a gentle slope is better, as it gives greater surface in contact with the tree, but still has enough slope to allow stacking. There should be a stout wire turned under the metal forming the rim, to give strength. Such buckets can be purchased in sizes of 8, 10, and 12 quarts. Some makers prefer the straight-sided buckets, and in such cases three sizes that easily nest, namely, 11, 13, and 15 quarts, should be bought.

These buckets may be obtained without handles. A round hole of an inch or less is made in the side just under the rim, and by means of this hole or a wire loop through it the bucket is suspended from the spout. If care has been exercised in the selection of the place of tapping the tree, there will be no need of driving a nail to keep the bucket in position.

One great advantage of the metal bucket is the ease with which it may be cleaned. All parts are easily reached and there is no danger of sap remaining to sour at a later date.

It is a good plan to paint the outside of new metal buckets in order to lengthen their time of usefulness. Painting them on the inside is not a very good practice as the paint film will soon come off, especially if the buckets are scalded or washed with hot water. If they are rusted on the inside to any extent, it is best to discard them. Iron buckets or an iron exposure will always change the sap, producing a dark-colored product; therefore the use of metal buckets does not assure a light-colored, good-flavored product. Care must be used with any kind to get good results.

COVERS.

The use of bucket covers is a disputed question. During the sap season rain and snow alternate with sunshine. Uncovered buckets hung on the trees are in a position to catch all this and, in addition, leaves, twigs, bark, insects, and dirt that may be swept through the air by the wind. The rain water dilutes the sap, besides often carrying with it the dirt from the trees as tree washings, which can not be removed from the sap; the only course is to empty the bucket. Twigs, leaves, insects, and such foreign material can be removed by straining the sap, but they leave a part of their soluble matter, which can never be removed. For these reasons one would naturally expect that the use of covers would become almost universal. Objections are made to their use in that more time is consumed in removing them when emptying the buckets; also that the sap sours more quickly in covered than uncovered buckets.

Makers who always use covers say that with a hinge arrangement fastened to the spout no extra time is necessary to empty buckets, and that if any extra time is used less time is needed in boiling the sirup and a better-flavored product is obtained, so there is an actual saving of time. By fastening the cover so that the back edge is raised a little above the front, a free access of air is possible. Many of the covers on the market are arranged in that way. Many advocates and users of covers refute the claim that the sap sours more easily when covered, asserting that sap sours rather from uncleanly methods. When little or no rain or snow falls during the sap season, there is not the need of covers. It is true, however, that darker sirup results from the uncovered bucket.



FIG. 5.—Open buckets. (Too many buckets on one tree.)

Covers on buckets may form a good guide to the maker in telling how often his buckets are emptied and whether they have been emptied on that day. This is accomplished by having the two sides of the cover painted different colors, as in one case noted one side was white and the other side red. As the sap was gathered the cover was turned. Thus, in passing through the bush for the afternoon collection the buckets of the morning collection could be passed. By this means a faithful worker will be sure all of his buckets are emptied each day.

GATHERING PAILS.

One of the largest buckets fitted with a handle may serve the purpose of collecting the sap from the buckets on the trees and carrying to the gathering tanks. The usual form is a metal pail with a larger diameter at the bottom than at the top. This gives it stability and

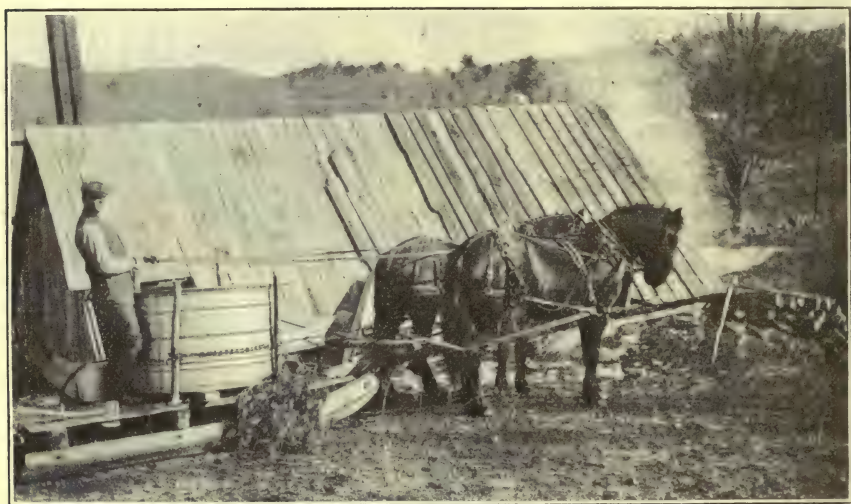


FIG. 6.—Hauling tank.

makes it less likely to turn over when resting the sap bucket on its edge to empty. Many have flaring funnel tops. As in the case of sap buckets, these might be of wood, but are unwieldy on account of their initial weight. They should be cleaned often, in fact after each time of using. The inside bottom edge, forming an acute angle, gives opportunity for dirt to collect and may soon become foul from bacterial growth. The gathering pails hold from 15 to 20 quarts.

HAULING TANKS.

Where only a few trees are tapped, and the boiling is done close by, the sap may be carried in the gathering pails to the supply tank for evaporation, but where the sugar bush covers many acres there is need of some means of transporting the sap. The usual form of transportation is in some large receptacle on a sledge or stone boat drawn by horses or cattle. Many of the small camps use a barrel

fitted with a funnel as the receptacle; this takes time to fill and also to empty when the sled is drawn up to the sugar house. Some set the barrel on end, knocking the head in and fixing a faucet at the other end for the sap to run out. Other makers use large wooden tanks, either round or square, fitted with an opening at the bottom and a standpipe (fig. 6).

There is the same objection to the use of these wooden tanks for collection as to the wooden sap buckets; but since the sap does not stay in them any length of time, the objection is not so strong. They should be painted on the inside before the beginning of the season and cleaned often when being used.

Many forms of metal tanks are made for this purpose. These forms are quite desirable on account of their ease of filling and emptying. The iron pipe at one end is loosely screwed onto the nipple at the bottom of the tank and is held upright by the lock at the top of the tank. To empty, the lock is taken out of the socket and the pipe turned downward. These tanks may be obtained in sizes holding from 2 to 6 barrels of 32 gallons each.

A flannel or cheese cloth may be stretched over the top of the tanks and the sap poured through this to remove any twigs, leaves, or pieces of dirt. This is of great importance in the production of a good grade of maple product.

BOILING HOUSES.

If the sugar bush is small and near the house or farm buildings, the boiling can be done in the summer kitchen, over the cookstove, or in a shed; but if 200 or more trees are tapped, some kind of a boiling house should be used and some attention paid to its location. In selecting a suitable place, the first point to consider is the distance from the sugar bush; it should be located in the bush or near by where no long hauls of sap are necessary and on a level spot where good drainage can be secured with higher ground near by to allow a drive where the gathering tank may be drawn up and its contents emptied by gravity. It is not a good plan, however, to locate a house close up against a high bank or cliff which would interfere with the draft for the chimney. If no low spot with a slight elevation is available, dirt can be piled up for the driveway.

A roof is not necessary, but it serves to house the sleds and apparatus, and in bad weather to keep snow and rain from the tank. After season this space is also available for the storage of the sap buckets.

The size and kind of sugar house must be regulated by the size of the camp or number of trees to be tapped. A convenient size is one in which there are at least 3 feet of space on both sides of the evaporator and 5 feet in front. The house should be at least 7 feet—and, better, 8 feet—high at the sides and covered with a well-slanting gable roof; a quarter pitch for the roof is recommended by many. There should be a ventilator as long as the evaporator in the ridge of the roof, and placed immediately over the evaporator. Many makers claim that a wooden roof is to be preferred to a metal one, as there is less condensation and dropping into the pan. Then, too, a metal roof without paint soon rusts and allows dirt to drop into

the pan. In connection with the house there should be a shed for the storage of wood to be burned in the evaporator.

It is not good policy to throw up a cheap, makeshift house, as the work is carried on in a trying time of the year, and the point of the health of the maker should also be considered. The sides of the

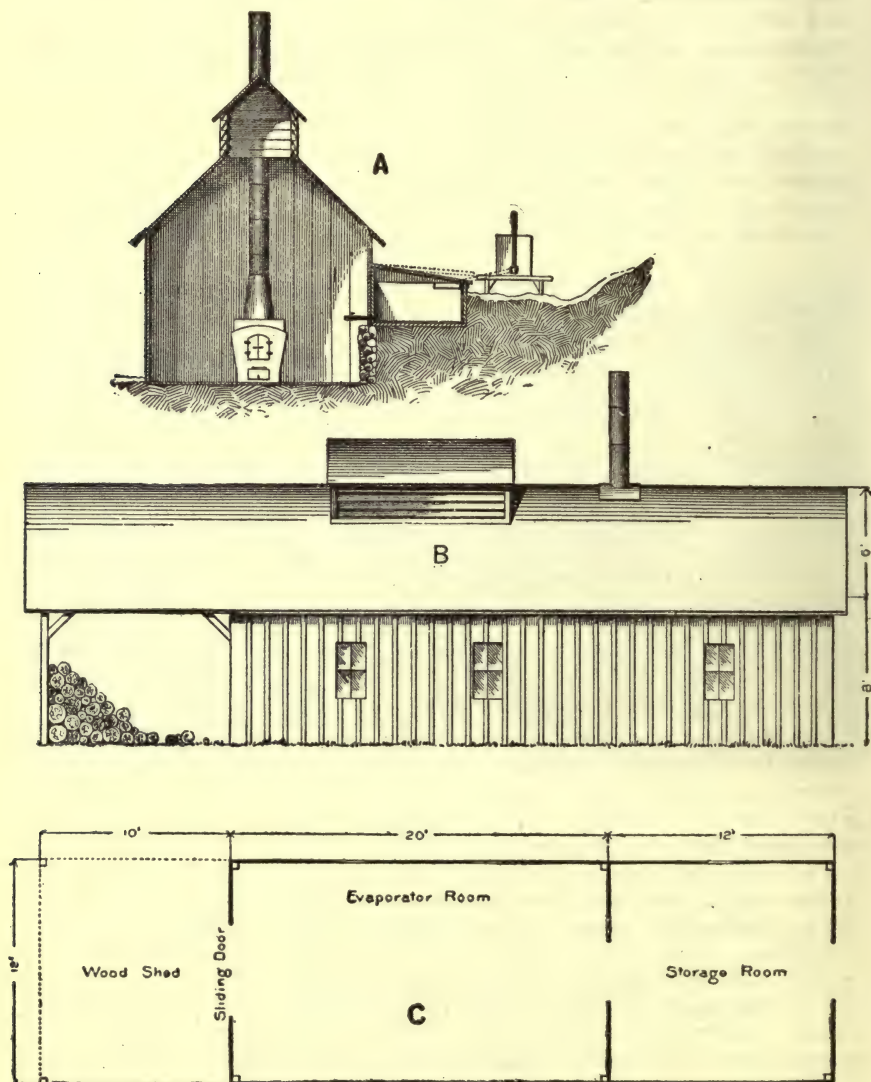


FIG. 7.—Plan of a model evaporation house: A. Sectional view, showing evaporator, storage tank, and gathering tank (on shed). B. Elevation. C. Ground plan.

house should be battened and all cracks covered over; the doors and windows should fit tight. Without these precautions the house soon becomes filled with partially condensed vapor or steam, and a cold, chilly dampness is noted, which greatly hinders evaporation. The

ventilator should be capable of being opened or closed easily; the floor should be of brick, cement, or wood, and should have a slope for ease of cleaning. Brick or cement is preferable to wood, because of less chance of fire. Figure 7 shows the plan of a sugar house which is said to have cost about \$125.

It is not necessary to have the small storage room as shown in C, but where sugar is to be made it forms a good place for the setting up of the "sugaring-off" pan (see p. 29). It is of special benefit to have the woodshed boarded up, but this is of less importance than the other portions of the house. The dimensions given allow for the ordinary sized evaporator, but for larger ones it may be found well to increase the width and length of the whole structure.

The storage tank should be a large one, capable of holding about a half day's run of sap. It can be made of wood, but it is better to be of metal. The wooden tanks soon fall to pieces and are constantly a source of loss through leaking. The tank should be



FIG. 8.—Iron kettle as an evaporator.

located outside the house and immediately against it. The bottom of the tank should be at least a foot above the top of the evaporator, to allow an easy grade for the tank to drain into the evaporator. This tank, which should be closely covered to exclude the rain and dirt, must be located on the side of the house next to the roadway, so that it is easily accessible to the gathering sled.

By partially burying the tank or boxing it in with wood and filling the space with leaves the sap will not freeze so easily in cold weather nor heat so soon in hot weather.

EVAPORATION APPARATUS.

Evaporation apparatus in general use may be divided into three general classes: (1) Iron kettles, (2) arch evaporators, and (3) patent evaporators.

The iron kettles are generally from 2 to 4 feet in diameter and are seldom housed. They are set on stones, as shown in figure 8, or suspended from a support. In some cases two or more are set in brickwork, with the place for the fire underneath. This form of evaporation apparatus is the crudest and seldom, if ever, yields a pleasant-tasting product. The danger of burning or scorching the sirup by the flames playing around the metal near the edge of the liquid is ever present.

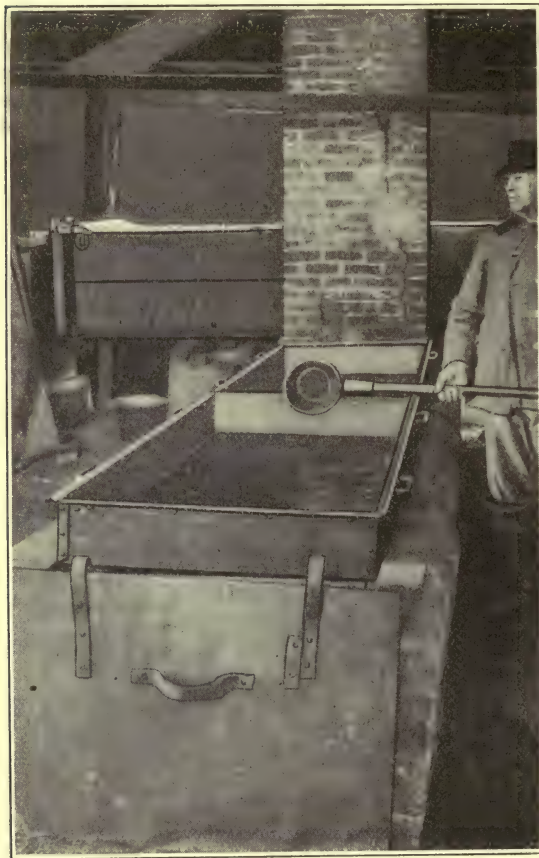


FIG. 9.—Arch with metal pans for evaporation; storage tank for sap in rear.

Another crude method of boiling is with sheet-iron pans, generally from 2 to 3 feet wide and 3 to 6 feet long. They are raised above the ground by a brick or stone work and the fire built under them. The same objection can be raised to them as to kettles, namely, the danger of scorching the sirup. They are, however, a step toward a better product. Many makers use pans made of sheet tin or galvanized iron. The former, with care in boiling, can produce a good-flavored product, but those of galvanized iron can not be recommended. Figure 9 shows such home-made pans in place. In this case there are two pans, one back of the other. The sap can be placed in the front pan and when

partially concentrated be transferred to the back one or vice versa by means of a ladle. The figure shows also the brick masonry constituting the arch. Because of this arrangement this form of concentrating apparatus is generally known as an arch evaporator. In most cases the arch is constructed of brick, but quite often stone is used. It may be 3 to 4 feet wide and from 8 to 15 feet long, inside measurements. The sides are generally from $2\frac{1}{2}$ to 3 feet high. Between these two walls the bottom is bricked or cemented and the walls are held together and in place by iron stays, which also serve to hold or support the pans. At the back the arch ends in

a flue and a brick or metal chimney extending through the roof. In the front grate bars may be provided and an excavation made for an ash pit.

In the arch and patent evaporators it is well to build the arch during good weather and allow it to set, as when built hurriedly before the season or in bad weather it is likely to crumble and break away. It is of extreme importance to see that the top layer of the two sides are of equal height so that the pan will set level; a slight slant of an inch in 16 feet in the level of the length of the arch is of value, as it gives the sirup a tendency to run toward the back where the final concentration takes place.

The pans are made to fit the arch and are not over 6 inches deep. The edges may be turned over a stout wire to give rigidity and strength, and should be fitted with handles so that they can be readily removed from the fire. The arch can be supplied with a regular stove front containing a fire door and ash door or be fitted as in the cut with a piece of sheet metal. The former gives a better and closer fitting front and allows a better control of draft.

Patent evaporators are simply improvements over the arch evaporators, but the fundamental principle is the same. The arch is generally metal, lined with fire brick. The pans fit the form much more closely to allow a better play of heat without loss, and often have corrugated bottoms to present a greater surface to the fire for quicker evaporation. The pans are also partitioned off to give a zigzag course to the sap.

Some forms have an apparatus at the side which automatically keeps a constant level of sap in the boiling parts and can be set and regulated to keep an even depth of boiling sap. The finished sirup can then be drawn off continuously.

The iron arch with its dampers allows the best results from the fire, which are hard to obtain in an ordinary arch built of masonry. Such patent evaporators can be obtained in sizes from 2 by 7 feet up to 6 by 24 feet. The larger sizes are capable of taking care of greater quantities of sap. In general, with this style of evaporator with corrugated bottom, 1 square foot of bottom is capable of concentrating about 2 gallons of sap per hour—that is, a pan 3 by 8 feet, or 24 square feet, will evaporate about 40 to 50 gallons of sap per hour and one 4 by 16 feet, or 64 square feet, will evaporate about 115 to 130 gallons per hour. Some makers have found that about 10 square feet of boiling surface are necessary for every 100 buckets set. That is, a camp of 500 trees would need an evaporator of 50 square feet. In figuring capacity, it is well to take the minimum figures rather than the average or maximum figures.

SUGARING-OFF APPARATUS.

Where maple sugar is made as a side issue or in very small quantities, it is customary to make the extra concentration in pots over the kitchen stove, but where made on a larger scale, special apparatus is used. Figure 10 shows a homemade sugaring-off pan. These pans are much shorter than the evaporators and very much deeper, generally from 2 to 2½ feet wide by 3 to 6 feet long and from 12 to

14 inches deep. The metal is usually heavy tin or in some cases galvanized iron, but never sheet iron. The sides slant toward the bottom and the edges are reinforced well with heavy wire. The illustration shows a faucet in the pan for drawing off the thickened sirup. The arch is made of brick with a fire box and ash pit. Where

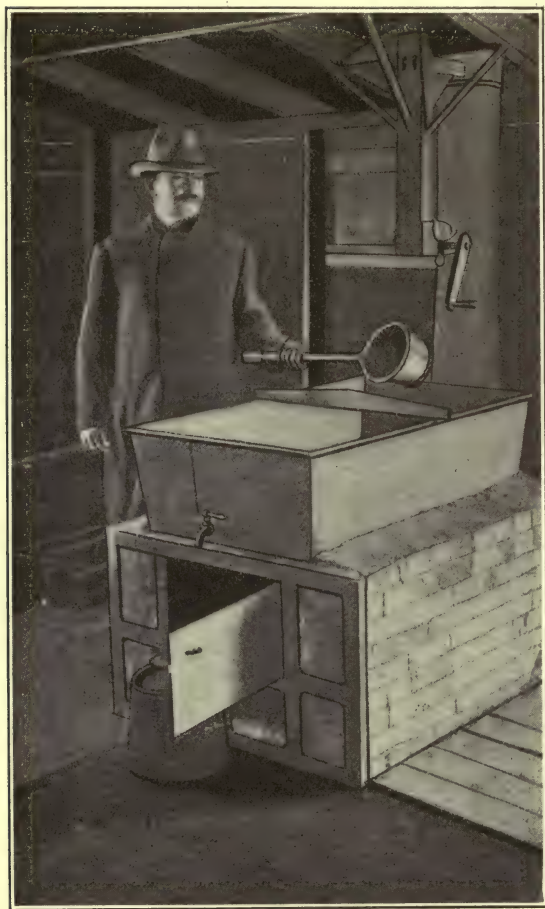


FIG. 10.—Sugaring-off outfit.

there is no means of drawing off the sirup the pan should be easily removed from the fire so that when the proper concentration has been reached the contents of the pan can be immediately poured out into molds.

It is well to have the sugaring-off pan in a separate room in the sugar house and located so that there are at least two rafters or stringers holding the sides of the house above the pan. By attaching block and tackle to these and then having four hooks on a ring and the ring attached to the block and tackle, the hooks can be inserted into the handles of the pan and one man can easily lift the pan from the arch and swing it to the part of the room where it is to be emptied. This is found extremely convenient.

Sugaring-off pans with metal arches can be obtained. They pos-

sess numerous points of advantage over the homemade pans, the main one being the absolute control of the heat by means of dampers.

MAPLE SAP.

The discussion up to this point has been altogether along the line of apparatus used in this industry. The sap and the methods of manufacturing it into sirup and sugar will now be considered.

The flow of sap is not continuous; that is, when the tree is tapped the sap does not start to run and continue until the season closes.

It runs only when the atmospheric conditions are favorable; as a rule the run is stronger during the day than at night and quite often in the middle of the day than in morning or evening. As the changes of weather occur the sap stops, to start again when the right conditions are present, so there may be a run of sap for a day or two and a lapse of a few days before another. During a season there may be as few as two or three runs or as many as ten or twelve. Freezing weather will stop the run, as will excessive wind, too bright sun, or a warm snap.

At the beginning of the season the sap is water white, clear, and transparent and has a sweet taste, but as the season advances the color and physical appearance change. It usually becomes cloudy and yellowish and gets a peculiar odor. The composition of the sap varies considerably, depending upon the season and the tree. There is anywhere from 1 to 5 per cent of solids in the sap, of which about 95 to 97 per cent are sucrose or ordinary sugar, so the sugar content varies anywhere from 1 to 4 per cent, with an average of 2 per cent or possibly more. Besides sucrose, there is present some nitrogenous matter and some mineral matter. Reducing sugars have never been found in normal sap by the author, although many hundreds of samples have been examined.

Containing as it does nitrogenous matter and also ash, the sap is very susceptible to the growth of microorganisms, which are believed to cause the souring of sap. Edson¹ has done much work along this line and shows that the change in physical condition of the sap toward the end of the season is due to a great extent to microorganisms. When the sap is running well the danger from them is not great, but when the warm weather starts and the flow is intermittent they become very active. Therefore the sap should be collected each day and not be allowed to accumulate. It is necessary also to keep the buckets and containers clean. After each run they should be washed with warm water. This is of extreme importance in obtaining a fine grade of sirup from the later runs. One of the visible signs of sour sap is the mucous formation in the buckets. When this is noted the buckets should be cleaned out thoroughly and scalded.

The tap holes as well as the buckets become infected. This infection increases more slowly in spouts with small openings than in hollow tubes with large bore. The latter allow a drying out of the wood tissue, which drying, together with the growth of microorganisms, slackens the flow of sap and causes what does flow to be a wrong color. It is under these circumstances that reaming of the tap holes is recommended, as this removes the dry contaminated tissue. At the same time the spouts should be steamed out or fresh ones inserted. This care in the sugar bush is necessary for a good product.

"Buddy sap" is the name applied to late runs of sap, especially that running at about the time the buds in the trees start to open. It is usually green in color or may be yellowish and has a peculiar odor easily recognized. Sirup produced from this sap does not have a good flavor and is always dark colored.

¹ Vermont Agr. Exper. Sta. Bul. 151; Bul. 167.

It has been popularly believed that this "buddy" condition is due to changes in the composition of the sap within the trunk of the tree due to the renewal of vegetative activity in the tissues, and that the alteration in color and flavor is a result of this change, but Edson¹ has demonstrated that this "buddy" flavor is due to a large extent to the action of microorganisms. When this peculiar odor or flavor is noted in the sap no more sugar or sirup is made.

Other conditions of sap are known as stringy sap, green sap, red sap, and milky sap. In the first the sap is ropy or stringy and generally milky in color, the second designates one with a greenish to greenish-brown color, the third shows a reddish sediment, and the last has a peculiar milky appearance. None of these saps produce a good sirup. Although they are spoken of as "sour saps," in reality they are not sour in the strict sense of the word, as the acidity is seldom much above the normal acidity of the sap, but they are simply changed by the microorganisms. It is claimed by Edson that when holes are running this peculiar sap, if the maker will rebore the tree some distance from the old hole and put in new sterile or clean spouts, normal sap will be obtained. The season may be prolonged to some extent by this procedure, but tapping more than the second time in one season is not to be recommended.

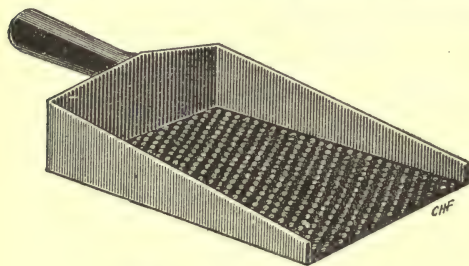


FIG. 11.—Sirup skimmer.

PROCESS OF MANUFACTURE OF MAPLE SIRUP.

CONCENTRATION OF SAP.

On frosty mornings ice is often found in the sap buckets and in some cases the whole mass of sap may be frozen.

Makers differ as to whether this ice should be thrown away. Where the whole quantity of the sap is frozen it is certain that it should not be thrown away, but where ice is floating the ice should be, as the freezing water would include very little of the sugar, and the sap would, therefore, be concentrated to some extent. This method of concentration is said to have been used by the Indians.

When the sap is heated a scum forms on the surface. This is the nitrogenous matter coagulating. Should there be fine sediment in the sap as it runs to the evaporator this will be caught in the coagulum and brought to the surface, when it should be carefully skimmed off.

Figure 11 shows a form of metal skimmer which resembles an ordinary dust pan with the bottom perforated. This may have a short handle, as shown, or a long wooden pole may be attached, by means of which the scum can be raked to the near edge of the pan or kettle and then lifted off. As the concentration goes on there is a deposition of mineral matter, which may float around in the thickening sap or may be deposited on the bottoms or sides of the

¹ Loc. cit.

pans. This mineral matter, commonly known as "sugar sand," "niter," "silica," is mainly a malate of lime. Its removal will be discussed under "Cleansing and straining."

In concentrating any sugar juice there are a few fundamental facts that should be known. Sugar in solution is easily broken up by long boiling, and as the solution becomes more concentrated the temperature of boiling is raised and more decomposition takes place. Then to retain the flavor of the thin juice in the resulting concentrated sirup, it must be evaporated as quickly and at as low a temperature as possible. The time consumed in evaporation can be regulated, but the boiling temperature can not be changed except by boiling in vacuo, which on a small scale or even on a rather large scale would not be possible, as the price of installation of apparatus would be prohibitive.

When using iron kettles they should be charged and this charge concentrated, the kettles being filled once or twice with fresh sap, but a light-colored, good-flavored sirup will not be obtained by keeping up the addition of fresh sap to the boiling kettle and only "siruping off" once or twice during the day. This concentrating, then diluting, and then concentrating induces decomposition of the sugar and organic matter and therefore blackens the sirup. It is difficult, as stated before (p. 28), to obtain a fine, mild-flavored maple product from concentrating in iron kettles.

Iron pans may produce dark-colored sirup, as also may patent evaporators if they are used to concentrate sap in the way the iron kettles are used.

The way to obtain good products with pans or patent evaporators, provided the sap is not sour, is to concentrate in as thin a layer as possible, drawing off the sirup as quickly as made. Where a large single pan is used this is hardly possible, but one can put in an inch or two of sap, boil, and add more, a little at a time, so as not to stop boiling or materially change the density of the boiling liquid; then, when this charge is concentrated, the sirup should be drawn off. Care must be exercised not to allow the remaining sirup in the pan to be burned. Where more than one pan is used the sap can be placed in the one over the fire, skimmed, and partially concentrated, then transferred to the next and further boiled, then ladled to the others, the last pan being the finishing pan. As one pan is emptied the sap from the one in front is brought to it. This means allows better skimming. In patent evaporators the sap and partially concentrated sirup flow through siphons from one compartment to the other, thus doing away with the use of a ladle.

To produce the highest quality, best-flavored sirup, the sap or semiconcentrated sap should not be deeper than 1 to 1½ inches in the evaporator at any one time. This produces a quick evaporation with the least quantity of decomposition.

CLEANSING AND STRAINING.

During concentration, as stated before, the mineral matter of the sap becomes more or less insoluble and precipitates out, a large portion incrusting the pan, and more or less floating in the semicon-

centrated solution and also in the finished sirup. This makes the sirup murky and less attractive in appearance. Settling removes a large percentage of this suspended matter, and if the semisirup is filtered through flannel a greater portion is removed.

Other means recommended for removing this sediment are white of egg, whole milk, and baking soda. These are added to the sirup in the last pan and work by coagulating around the suspended matter and bringing it to the top or by liberation of a gas, causing foam to appear. Then, by careful skimming, this is removed. When used in quantities these settlers change the flavor of the sirup and, contrary to the general belief, do not render the sirup lighter in color. Baking soda, added to the finishing sirup to make it foam and bring the foreign material to the top, also tends to neutralize any acidity. Its use is a very questionable practice, for if in any quantity it darkens the sirup besides greatly changing the flavor.

This cleansing can be accomplished by allowing the finished sirup to settle or by pouring off the sirup from the evaporator through felt or flannel into the cooling cans, and as a rule this will give as light-colored sirup as when the clarifiers are used. Felt strainers shaped like caps can be obtained. Such a strainer may be 10 inches in diameter at the top and 14 inches deep. By suspending this in the neck of a large milk can and turning the edges the hot sirup readily filters through. By having cocks arranged in the settling can, the clear sirup can be drawn off from the top cock after the sirup has been allowed to stand, and then be drawn from the lower ones in turn, stopping when the sediment is reached. Double thickness of heavy flannel serves as a fair substitute for the felt. The sirup should be filtered hot, as it is very difficult to get it to pass through when cold.

DETERMINATION OF FINISHING POINT.

Commercial maple sirup weighs 11 pounds to the United States gallon. Such a sirup will have 65 per cent solids or 35 per cent water. A sirup made thinner than this—that is, containing more water—will soon sour, and one made much thicker—near 12 pounds to the gallon—will tend to crystallize. The maker has to determine the density of his product. This is often accomplished in a crude way by noting the bubbles as they break on the surface of the boiling sirup or by removing some in a spoon and watching how it pours. A more satisfactory way is either by means of a thermometer to determine the boiling point or with a Baumé hydrometer spindle to determine its density.

By thermometer: The boiling point of a liquid is influenced by its concentration and also by the altitude. Water at sea level boils at 212° F., but for every 500 feet above sea level, roughly speaking, the boiling point is lowered 1°. Therefore, at an altitude of 2,000 feet the boiling point of water is 208° F. The Vermont Agricultural Experiment Station has found that a maple sirup boiling at 219° F. weighs 11 pounds to the gallon, or is at standard density. This figure, however, changes a little, depending on the run of the sap. A first run will often boil at 217° or 218° F.

when having a concentration equal to 11 pounds to the gallon. It is necessary, then, to note the boiling point of the sirup only in the last pan to decide whether it is concentrated sufficiently. In taking this temperature, one must be sure the thermometer is correct and also that it does not touch the bottom or sides of the evaporator, but measures the temperature of the liquid only.

When a thermometer is used, if the temperature which it registers when placed in the boiling water is noted, and 7° added to this, the result will be the point at which the sirup boils when it is properly concentrated. It is of extreme importance, however, to test the

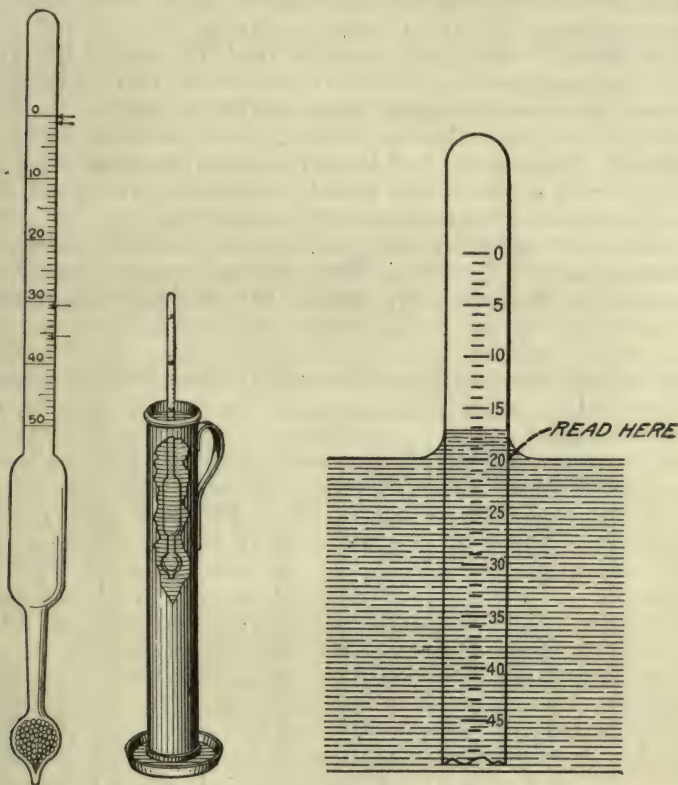


FIG. 12.—Hydrometer and its position in the liquid.

sirup again by weighing a quart or a gallon of the same after it has cooled to make sure that it has a proper density.

By Baumé hydrometer: A hydrometer or spindle is an instrument for showing the density of a liquid. Hydrometers are graduated to various scales and for various purposes. The one generally used for rough sugar work is the Baumé. The standard of graduation is an arbitrary one and varies somewhat with different makes. The usual Baumé hydrometer is made of glass and shows a graduation from zero to 50, divided into degrees, as shown in figure 12. The density is measured by floating the hydrometer freely in the liquid, which is generally held in a tall cylinder, as shown in the illustration. The

point on the scale where the instrument comes to rest is considered the density. It will be noted that the surface of the liquid is curved up at the points of contact with the metal cylinder, and also with the hydrometer. The correct reading of the instrument is on a line with the surface of the liquid as shown in the cut and not at the upper edge of the curved portion. The temperature at which Baumé hydrometers are standardized is 60° F., unless it is otherwise marked on the stem of the hydrometer, so for correct readings the sirup should be cooled to that temperature. A sirup of standard density has a Baumé reading of 35.6°, but if measuring the density of the boiling liquid by drawing some out into a cylinder the density will be somewhere about 32° to 34° and on cooling 35.6° to 36°. This is readily understood when one considers that the zero of the hydrometer is at the upper end and the 50 at the lower end. On heating a liquid, there is an expansion and the liquid is lighter, so the hydrometer sinks farther down than on cooling, when there is a contraction of the liquid. The accuracy of these glass hydrometers is very much affected by using them in hot liquids, hence it is not good practice to use the accurate instruments in the hot sirup.

The following table shows the solid content and the water content for different degrees Baumé. These figures are only approximate. A gallon of the finished sirup should be weighed to give accurate results.

Dry substance and water corresponding to each degree Baumé.

Degrees Baumé. ¹	Dry substance.	Water.	Degrees Baumé. ¹	Dry substance.	Water.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
1	1.7	98.3	26	46.8	53.2
2	3.5	96.5	27	48.6	51.4
3	5.3	94.7	28	50.5	49.5
4	7.0	93.0	29	52.4	47.6
5	8.8	91.2	30	54.3	45.7
6	10.6	89.4	31	56.2	43.8
7	12.3	87.7	32	58.1	41.9
8	14.1	85.9	33	60.0	40.0
9	16.0	84.0	34	61.9	38.1
10	17.7	82.3	35	63.9	36.1
11	19.5	80.5	36	65.8	34.2
12	21.3	78.7	37	67.8	32.2
13	23.0	77.0	38	69.7	30.3
14	24.8	75.2	39	71.7	28.3
15	26.6	73.4	40	73.7	26.3
16	28.4	71.6	41	75.7	24.3
17	30.3	69.7	42	77.7	22.3
18	32.1	67.9	43	79.7	20.3
19	33.9	66.1	44	81.8	18.2
20	35.7	64.3	45	83.8	16.2
21	37.5	62.5	46	85.9	14.1
22	39.4	60.6	47	88.0	12.0
23	41.2	58.8	48	90.1	9.9
24	43.1	56.9	49	92.2	7.8
25	44.9	55.1	50	94.4	5.6

¹ Taken at 60° F.

It is not to be understood that a degree Baumé corresponds to 1.7 per cent of sugar, for the hydrometer measures other dissolved solids also.

In order to determine relatively the number of degrees Baumé of the cooled sirup when the reading is made with the hydrometer at

a high temperature, it is necessary to take the temperature at the same time that the hydrometer is read. Subtract 60 from the number of degrees Fahrenheit of the heated sirup (this being the normal temperature) and multiply the difference by 0.0265. This figure (which is the temperature correction expressed in degrees Baumé) is added to the Baumé reading of the hot sirup and the result is the Baumé reading of the cooled sirup. For example, a heated sirup shows a reading of 30 at a temperature of 215° F. Then—

$$\begin{aligned} 215 - 60 &= 155 \\ 155 \times 0.0265 &= 4.1 \\ 30 + 4.1 &= 34.1 \end{aligned}$$

The cooled sirup would read 34.1 or by above table have about 38 per cent water.

CANNING AND STORAGE.

After the sirup is strained or settled, it is ready for canning. Makers vary as to whether it should be canned hot or cold. Some claim that canning hot tends to induce crystallization. It is certain that when canned hot in sterile cans (steamed or washed in boiling water) there is practically no danger from fermentation. This can not be said of canning cold, although the sirup made one season is generally sold before the beginning of the next season. When made on a large scale, maple sirup is often run direct from the evaporators into barrels and so shipped, but by far the greatest number of sirup makers sell in quart, half-gallon, or gallon tin cans. These cans are generally square, with a screw cap. When being filled they are tipped slightly and then lifted by the upper edges and filled even with the screw top, which is then fastened tight with a wrench. This method applies to canning hot or warm sirup. When the cans are being filled in cold weather and with cold sirup, it is well to hold the can so that the sides are a little compressed and then fill to the top and screw the lid on. By this means no air enters and when the sides are released the can being not quite full allows for increase in volume when placed in a warmer room, without danger of breaking. Fancy maple sirup is often put up in glass and when carefully canned will keep from one season to another without souring or bursting the jar. It is best to store sirup at an even, cold temperature. Temperatures around freezing, however, should not be used, as this tends to crystallization.

CRYSTALLIZATION.

Crystallization results from making a supersaturated solution of a substance in a liquid. In the case of sirups the sucrose becomes concentrated to a point at which it is no longer soluble in the water present, and hence it crystallizes out. Maple sap, being a dilute solution of sucrose, in fact about 90 to 95 per cent of the solids in the sap being this sugar, when concentrated soon reaches a point where the sucrose will be supersaturated, and hence on cooling will start to crystallize out.

Pure water will hold in solution 66 per cent of pure sucrose at ordinary temperatures. If there is a concentration by evaporating off the water to a point where there is 66 per cent of sucrose in the resulting solution, the solution is holding all the sucrose it can without depositing some as crystals. In practice, crystallization sometimes occurs before this 66 per cent is obtained. The commercial standard gallon of sirup, weighing as it does 11 pounds to the gallon, contains at least 65 per cent solids, of which at best 95 per cent is sucrose, and should not crystallize. If, however, the resulting sirup weighs 12 pounds to the gallon, there is much more chance of crystallization.

Makers vary as to the question of the influence of hot and cold canning on the possibility of crystallization. Some claim that by canning hot crystallization is prevented, others vice versa. It seems, however, that it is more a question of the density of the product than of the heat of canning. These crystals may form in small patches or may be large individual ones. Their presence in maple sirup is often considered by some purchasers as an evidence of adulteration by addition of rock candy, which is not true to fact. Once crystallization is started, these crystals grow until solution equilibrium is attained.

Crystallization is induced in many cases by changes in temperature, the solutions becoming cold and then warm, hence it is well to store maple sirup in a place of as even temperature as possible. The first runs are more likely to show crystallization than the later ones.

MANUFACTURE OF SUGAR FROM MAPLE SIRUP.

"SUGARING-OFF."

"Sugaring-off" applies to the further treatment of the maple sirup by which it is made into a solid product. In careful manufacture of sirup, even from sour sap, a solid product may be produced by concentrating to a higher degree. But if during manufacture cleanliness has not been regarded or sour sap has been used, a solid product will not result from any amount of boiling, due to the large amount of inversion of the sucrose.

The sugar, sucrose, which constitutes about 95 per cent of the solids of the sap and, after evaporation to sirup with care, constitutes from 90 to 96 per cent of the solids of the sirup, is easily broken down or split up into two sugars known as dextrose and levulose. The former of these crystallizes easily, while the latter does not. It is the formation of these sugars from sucrose that tends to prevent the making of sugar from certain sirups. This is also the reason why late runs of sap or burned sirup or sour sirup will not yield sugar and also why some sirups will produce a hard sugar while others produce only a soft mushy sugar provided the finishing-off points are right.

Where maple-sugar making is conducted as a side line to the sirup making, the ordinary iron pot of the kitchen is filled nearly half full with the sirup and this concentrated over the kitchen

fire, but where on a larger scale the sugaring-off pan (p. 29) is used. In this concentration there is a further precipitation of the malate and also a great tendency for the boiling product to froth or foam. A small piece of butter or lard or some sweet oil or a piece of fat meat, as bacon or fresh pork, run over the surface is used to allay this foaming. These may be necessary to allay the foaming at times, but great care should be used in the selection of the fat to have a neutral one with little taste or flavor and then to use it only in very small quantities, as the flavor of the product is greatly affected. If the precipitation of malate is rather heavy, the white of egg or milk may be added and this skimmed off during the boiling. Under any circumstances the boiling mass should be skimmed.

The hardness of the sugar produced is to a large extent controlled by its moisture content; also slightly by the inversion of the sucrose during the heating. As in sirup making one should "sugar-off" a charge before adding any more sirup, as this concentration and dilution and concentration again tends to blacken the product and decompose the sucrose, making hard-sugar production difficult. The proper point of stopping the boiling is best determined by means of a thermometer. Various makers have other means of determining this point, such as by dropping some of the liquid in cold water or snow, but where a product of uniform hardness is to be prepared the former method is to be preferred.

In making maple sugar some consideration must be taken of the grade and use, as, for instance, maple sugar for immediate use should be softer than that for storage and cake sugars are generally harder than tub sugar. In the first runs the boiling should be carried up to 238° or 240° F. (or 26° to 28° above the boiling point of water at that elevation) to make a medium hard sugar, while for a tub sugar (one that is sold in tubs) a temperature as low as 233° F. (21° above boiling point) can be used. With later runs of sap the finishing temperature should be 240° to 250° F. (or 28° to 38° above the boiling point). These are not absolute in all cases. The maker can experiment with the proper temperature, but to obtain a uniform product it is necessary to use a thermometer.

As to the quantity of sirup that should be "sugared-off" at one time, makers vary. Some "sugar-off" as much as 50 to 100 pounds (4½ to 9 gallons) at a time, while others only from 15 to 25 pounds (1 to 2½ gallons), the claim being that with the larger quantities the sugar is made much darker, due to the longer time consumed in boiling or concentrating.

After the thick sirup has reached the proper boiling point, it should be taken from the fire and stirred until somewhat cooled. This gives it a uniform grain and color in the mold. The finishing temperature can be made a little lower if there is vigorous stirring during the cooling period, as by this stirring much evaporation takes place. If it is not stirred the point of quickest cooling, namely, the edges, becomes hard and coarse grained and the center or part last cooled is mushy.

MOLDING.

Wooden molds are used almost altogether for large-sized cakes and tin for the smaller ones. A very convenient kind for large-sized cakes is a wooden box with the sides clamped, so that when the sugar is molded the sides can be removed. By having the sides grooved, sheets of wood or metal can be inserted and smaller cakes made from the same mold.

The mold should be dry and warm. If the mold is wet, the cake of sugar will not be of uniform color; if cold, the cake is likely to stick at the sides, causing the sugar to harden there and be soft on the inside.

GRAINED OR STIRRED SUGAR.

Grained maple sugar derives its name from the fact that it is concentrated to a high degree, then stirred continuously during cooling. A mold is not used. The finished product is, as a rule, dry and somewhat lumpy, resembling the ordinary commercial brown sugar in appearance, but, of course, retaining its maple taste. In color it varies from a nearly white to a dark brown. It has been used as a table preparation.

STORAGE OF MAPLE SUGAR.

Like brown sugar, maple sugar does not keep well in a moist atmosphere. It tends to absorb water, molds rather quickly, and if finished at too low a temperature the sugar is soft and the liquid portion drains out. Therefore sugar which is to be stored should always be boiled to a high temperature. After being taken from the molds it can be wrapped in paper, but should not be put in covered containers unless these are absolutely sealed. It is best to store the sugar in a warm room of even temperature. If the cakes are sealed without access to air, a cold place can be used, but in no case should the storage room be damp.

ECONOMIC CONSIDERATIONS.

CARE OF APPARATUS.

When the buddy sap has started to run and no more high-grade maple products can be produced it is time to close the season of manufacture. The buckets should be removed from the tree and stood upside down on the ground to dry in the sunshine. The better makers wash them, while many claim that the last sap is good to leave in the buckets, as it prevents rusting. After they are thoroughly dry they should be stacked in piles and put in the sugar house, barn, or other shelter away from the rain. In stacking, rope or straw should be placed between the buckets, so that they will not fit tight in one another. This method is of great assistance when taking the stack down at the beginning of the next season. Some buckets have a bulged circle around them, which does not allow the buckets to become wedged together in stacking. After the spouts have been removed, care being taken not to break the bark of the

tree, they should be boiled in water once or twice, then allowed to dry thoroughly in a warm place before storing them in a box. The collecting buckets, hauling tank, and storage tank should be painted on the outside, and if of wood on the inside.

The evaporator should be cleaned out, the ashes removed, and a coat of asphalt paint or red iron paint given to the exposed metal parts.

The scale in the pans is best removed by boiling water in them and then using a fine wire brush. The scale, known as "sugar sand," "silica," or "malate," is a nearly pure malate of lime, the mineral matter of the sap that on concentration has been deposited in the pans when its solubility in the liquid has been reached. It has no particular economic use; some use it for scouring, and attempts have been made to use it for the preparation of malic acid. During the manufacturing season this scale should be removed from the pans after each boiling, as it hinders the boiling and requires more fuel to do the boiling.

Other methods of removing the scale are by scraping, by adding muriatic acid (hydrochloric acid) or vinegar, or by using butter-milk. Scraping weakens the pan and is not to be recommended and acids or vinegars should be used with the greatest care, as the metal of the pan may dissolve along with the scale.

After removing the scale, the pans should be washed out, thoroughly dried on the inside, and painted on the outside. They should be stored in a dry place, and turned over so as not to collect dirt or water during the summer. If a metal smokestack is used, this should be taken down, freed from soot, painted, and after drying, be stored in a dry place.

If the arch shows cracking or sagging repairs should be made during the following spring or summer rather than at the beginning of the next season. Care along these lines will preserve utensils, and they will be ready for use on a day's notice in the early spring.

YIELDS.

No very definite data can be given on the yields from the maple industry as so many factors enter into the subject. From 5 to 40 gallons of sap are obtained from a tree during a season; an average is somewhere between 10 and 20 gallons. Normal sap of an average year contains about 2 per cent of sugar, although it may vary from 0.5 per cent to as high as 7 or even 10 per cent. The sugar content varies greatly with the tree, its location, and its past growth. From one tree one can count on from 1 to 7 pounds of sugar per season, or expressed in sirup of standard density, from 1 pint to 1 gallon, though the average from year to year and from tree to tree is about 3 pounds of sugar or 3 pints of sirup. Expressing these data in other terms, it is seen that in a normal year 1 barrel of sap (32 gallons) should produce a gallon of sirup or $7\frac{1}{2}$ pounds of sugar. In many camps and for many years it takes sometimes as high as 50 gallons of sap to make 1 gallon of sirup. First runs of sap are generally richer in sugar, hence take less for a gallon of sirup. From $6\frac{1}{2}$ to 9 pounds of sugar, according to the kind, can be made from 1 gallon of standard sirup, with an average of $7\frac{1}{2}$ to 8 pounds.

A camp of 100 trees should produce about 40 gallons of sirup or 300 pounds of sugar.

COST OF MANUFACTURE AND SELLING PRICE.

The cost of manufacturing maple products is an extremely variable figure. The season of production comes at a time of the year when little or no other work can be done on the farm, thus allowing the aid of the family and farm help for the boiling and manufacture. Moreover, since the sugar bushes as a general rule are situated on hilly country that would not be suitable for any other crop, these two items could hardly be placed at a high value in a table of costs. The individual trees of some bushes average more than those of others, and some makers use more care and obtain better yields, so, all things considered, one can hardly place an actual estimate on the cost of manufacture. Makers on a large scale claim that the cost is from 45 to 75 cents a gallon for sirup, and from 5 to 8 cents a pound for sugar. With small makers, the cost tends toward the lower limit if no help is employed, and toward the upper if help is employed. It is claimed that one man and one boy can run a camp of 500 trees and manufacture the product. There is a question, however, whether a good grade product can be produced with so little labor, as one person has to be at the evaporator all the time when the sap is boiling. A poorer grade results from loading up the evaporator and going away to collect the sap.

Ohio makers who have taken up this question of cost of manufacture believe that at 50 cents a gallon there is no profit. There have been seasons of plenty, however, when the makers were offered only 45 cents a gallon in barrel lots, and many claim they made money even at that low figure.

The grade of product is an item in the cost of manufacture. Sirup requiring only the concentrating of the sap to a standard density costs less to manufacture than sugar which requires extra evaporation. It is claimed that sirup sold in bulk at 50 cents a gallon is equal to sugar at 7 cents a pound, at 58 cents a gallon is equal to sugar at 8 cents a pound, at 66 cents a gallon is equal to sugar at 9 cents a pound, and at 74 cents a gallon is equal to sugar at 10 cents a pound, but this is hardly true as it costs at least 5 cents a gallon to turn the sirup to sugar.

The cost of placing the goods on the market varies; in the case of sirup, screw-cap cans are generally used in sizes of pint, quart, and gallon, and in the case of sugar, the fancy cake, the large brick, and the tub sugar are the usual forms.

The question as to whether the production of sirup or of sugar is more profitable can be decided only by knowing the market where they are to be sold. Maple sirup in gallon cans can be sold at from \$1 up to \$2, while in barrels as low as 45 cents has been paid for it. Seventy to seventy-five cents, however, is a better average price for sirup in barrel lots. Sugar sells anywhere from 7 to 25 cents a pound, depending on the grade and form. The highest price is obtained for maple sugar put up in small, 1 or 2 ounce cakes, while possibly the lowest is for brick and tub sugars. An ordinary price is about 10 to 15 cents a pound.

FARMERS' ASSOCIATIONS.

It is practically impossible to produce a grade of sirup of uniform flavor and color. The run of sap and manufacturing conditions are the principal factors influencing these qualities. A maker often finds it impossible to make the same grade on different days with practically the same run of sap; therefore to have uniformity one should mix the various lots into two or more standard grades. A purchaser does not know why there are such differences in the product and wants two successive purchases of the same brand to be of the same grade; one desires the mild, sweet, maple flavor, while another wants the stronger flavor, and in order to satisfy these demands and at the same time build up a business a number of makers could cooperate to ship their products to a certain point and then reship. This is actually being done in Vermont, and after many years' trial it has been found to be of great advantage to the individual maker.

It is to be regretted that selling in large amounts is not done more on a sliding scale, depending on color and flavor. Many organizations and buyers offer better prices for the lighter-colored and milder-flavored product, thus stimulating producers to more cleanly methods of manufacture and so to a better profit in the business. It seems safe to say that this sliding-scale method is coming more into use, for without it there is no incentive for making the better grades of sirup and sugar.

ACCESSORIES TO MANUFACTURE.

FUEL.

Fuel is an important item in manufacture. It is usually wood, although sometimes supplemented by coal. Before the winter sets in the woodshed mentioned in the description of the boiling house (p. 27) should be filled with cordwood and small brush, as green wood does not produce a hot fire and requires too much attention from the man attending to the evaporator. Wood cut in the spring and piled under cover for the next spring's work will be dry and good. The amount that will be needed depends on the kind and dryness. Some makers state that about 8 cords of wood are necessary for 500 trees; last season one man used 10 cords of wood and 5 tons of coal for manufacturing sirup from 1,000 trees, and another used 35 cords of wood with 4,000 trees.

LABELS.

In preparing labels, there is one point possibly not well understood: that is, the distinction between sap sirup and sugar sirup. Maple sap sirup results from the concentration of maple sap with or without the usual clarifiers to a standard density. Maple sugar sirup is the solution to a standard density of maple sugar which has come from a further concentration of sap sirup. It is then not correct to label a product as a sap sirup which has resulted from the solution of maple sugar nor as a sugar sirup when made from sap. Both are maple sirups. As a rule sap sirups are sweeter, pleasanter to taste, and milder than sugar sirups, and possess a peculiar inde-

scribable property of the sap which is lost when sugar is made and redissolved.

A suitable paste for paper labels on tin cans is prepared from—

Silicate of soda-----	1 ounce.
Cornstarch-----	1 ounce.
Cold water-----	1½ pints.

Add the starch and silicate to the water and stir until the whole is smooth, then place the vessel in another one containing a little water and heat until the starch is gelatinized. This paste should be made often, as it soon loses its sticking properties.

PAINT.

Any paint will serve for the outside of sap buckets, gathering pails, and storage tanks. The ordinary red paint of an iron oxid base is one in common use. It is often difficult to make paint stick to metal, because of the grease on the surface, but this difficulty can be removed by washing the metal surface with soap and water or a little benzin and drying it before the paint is applied. Farmers' Bulletin 474, on The Use of Paint on the Farm, gives details regarding this and other paints and should be consulted. For the inside of such apparatus, where the sap comes in contact, it is a questionable procedure to use white-lead paints on account of the solubility of the lead. Some form of enamel would be better, but the price may prohibit its use. The iron paint already mentioned will serve admirably for this purpose.

Sap buckets should be painted in the summer or fall and then thoroughly dried before being set away. Never paint just before opening the camp, as metal buckets freshly painted inside have a marked tendency to taint the sap. There is the same objection to freshly painted wooden buckets, but in this case it will not last as long.

For the ironwork of the evaporator nothing better can be recommended than an asphalt paint or the ordinary iron paint, which should be applied after thorough scraping at the close of the season. Its use will greatly lengthen the life of this apparatus.

STATISTICS OF THE MAPLE SUGAR AND SIRUP INDUSTRY OF THE UNITED STATES.

It is a rather difficult task to obtain figures for the actual amount of maple products produced in the United States, first, because the products are made in small quantities by many farmers, and, second, because a large part of them does not reach the market, but is sold to friends of makers. The census reports for the years 1850 to 1910 give figures for maple production, sugar only having been reported in 1850, while in the other years sirup also was given. These have been arranged in order of their importance in production—first, maple sirup; and, second, maple sugar, as reported by the 1910 census, in which year reports from 79,381 farms on maple sirup and 29,444 farms on maple sugar were received.

Maple sirup production of the United States.

(U. S. Census Reports.)

[In gallons.]

No.	State.	1910	1900	1890	1880	1870	1860
1	Ohio.....	1,323,431	923,519	727,142	495,839	352,612	370,512
2	New York.....	993,242	413,159	457,658	266,390	46,048	131,843
3	Vermont.....	409,953	160,918	218,252	128,091	12,023	16,253
4	Pennsylvania.....	391,242	160,297	154,650	140,667	39,385	114,310
5	Indiana.....	273,728	179,576	180,702	242,084	227,880	292,908
6	Michigan.....	269,093	82,997	197,775	131,990	23,637	78,998
7	Wisconsin.....	124,117	6,625	48,006	58,012	31,218	83,118
8	New Hampshire.....	111,500	41,588	81,997	79,712	16,884	43,833
9	Massachusetts.....	53,091	27,174	33,632	13,017	2,326	15,307
10	Maine.....	43,971	16,024	71,818	82,006	28,470	32,679
11	West Virginia.....	31,176	14,874	19,032	28,696	20,209	
12	Illinois.....	18,492	9,357	13,978	40,077	10,378	20,048
13	Minnesota.....	17,808	1,079	12,091	11,407	12,722	23,038
14	Maryland.....	12,172	5,825	1,021	2,043	374	2,404
15	Missouri.....	9,389	5,474	8,333	16,244	16,317	18,289
16	Iowa.....	8,596	2,662	14,413	17,766	9,315	11,405
17	Virginia.....	6,046	1,677	3,468	7,518	11,400	99,605
18	Connecticut.....	4,236	948	1,437	2,173	168	2,277
19	Kentucky.....	3,547	2,367	10,468	27,530	49,073	140,076
20	New Jersey.....	504		134	334	5	8,088
21	North Carolina.....	404	129	1,142	582	418	17,759
22	Tennessee.....	373	171	1,186	3,688	4,843	74,372
Total ¹		4,106,418	2,056,611	2,258,376	1,796,048	921,057	1,597,589

¹ The totals include small amounts not reported under individual States.

The amount of sirup made in the individual States varies somewhat from year to year. Ohio leads in the six years for which the figures are given, New York standing second except in 1870 and 1860. Indiana stands third in two years, being second in 1860 and 1870 and fifth in 1890 and 1910. Vermont is fourth in 1900, third in 1890 and 1910, and sixth in 1880, falling far short of this in the other two years. The variation in these figures is influenced partly by the relative production of maple sugar, as some States use much more sap for sugar than others.

Maple sugar production of the United States

(U. S. Census Reports.)

[In pounds.]

No.	State.	1910	1900	1890	1880	1870	1860	1850
1	Vermont.....	7,726,817	4,779,870	14,123,921	11,261,077	8,894,302	9,897,781	6,349,357
2	New York.....	3,160,300	3,623,540	10,485,623	10,693,619	6,692,040	10,816,419	10,357,484
3	Pennsylvania.....	1,188,049	1,429,540	1,651,163	2,866,010	1,545,917	2,767,335	2,326,525
4	New Hampshire.....	558,811	441,870	2,124,515	2,731,945	1,800,704	2,255,012	1,298,863
5	Maryland.....	351,908	264,160	156,284	176,076	70,464	63,281	47,740
6	Michigan.....	293,301	302,715	1,641,402	3,423,149	1,781,855	4,051,822	2,439,794
7	Ohio.....	257,592	613,990	1,575,562	2,895,782	3,469,128	3,345,508	4,588,209
8	Massachusetts.....	156,952	192,990	558,674	878,793	399,800	1,006,078	795,525
9	West Virginia.....	140,060	141,550	177,724	310,866	490,066		
10	Virginia.....	44,976	19,310	26,991	85,693	245,093	938,103	1,227,665
11	Kansas.....	40,016				938	3,742	
12	Indiana.....	33,419	51,900	67,329	235,117	1,332,332	1,541,761	2,921,192
13	Wisconsin.....	27,199	4,180	128,410	488,837	507,192	1,584,451	610,976
14	Maine.....	15,388	5,500	84,537	153,334	160,805	306,742	93,542
15	Missouri.....	11,638	12,055	20,182	58,964	116,980	142,029	178,910
16	Minnesota.....	11,399	29,580	34,917	76,972	210,467	370,669	2,950
17	Kentucky.....	10,697	2,340	11,259	66,535	269,416	380,941	437,405
18	Connecticut.....	10,207	4,930	8,617	44,092	14,266	44,259	50,796
19	Iowa.....	6,173	2,320	45,120	50,710	146,490	315,436	73,407
20	Illinois.....	5,366	4,090	13,260	80,193	136,873	134,195	248,904
21	Tennessee.....	4,326	1,160	9,167	31,296	134,968	115,620	158,557
22	North Carolina.....	3,305	1,180	7,713	4,103	21,574	30,845	27,932
23	New Jersey.....	1,195		210	2,496	419	3,455	2,197
Total ¹		14,060,206	11,928,770	32,952,927	36,576,061	28,443,645	40,120,205	34,253,436

¹ The totals include small amounts not reported under individual States.

As noted with sirup, the production by States varies somewhat from year to year. Vermont stands first in all the years reported but 1860, when it was second to New York. New York is second in all the years except 1860. According to these figures, these two States produced over 77 per cent of the total crop in 1910, over 70 per cent in 1900, over 74 per cent in 1890, over 60 per cent in 1880, over 54 per cent in 1870, and nearly 50 per cent in 1860 and 1850. These two States, together with Pennsylvania, Ohio, New Hampshire, Maryland, and Michigan, have produced over 90 per cent of the total maple sugar since 1880.

The money value of the maple (sugar and sirup) industry for 1910 was \$5,177,809. The rank of the States is as follows: New York, \$1,240,684; Ohio, \$1,099,248; Vermont, \$1,086,933; Pennsylvania, \$417,213; Michigan, \$333,791; Indiana, \$300,755; and New Hampshire, \$182,341.





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